



Trimethylbenzene (mixed isomers)

Method number: 1020

Version: 1.0

Target concentration: 25 ppm
 OSHA PEL: 25 ppm (120 mg/m³) construction and shipyard
 ACGIH TLV: 25 ppm (123 mg/m³)

Procedure: Active samples are collected by drawing workplace air through charcoal tubes with personal sampling pumps. Diffusive samples are collected by exposing SKC 575-002 Passive Samplers to workplace air. Samples are extracted with 99/1 (v/v) carbon disulfide/*N,N*-dimethylformamide and analyzed by gas chromatography (GC) using a flame ionization detector (FID).

Recommended sampling time: 240 min

Sampling rates:
 226-01 Anasorb CSC 50 mL/min (12 L)
 SKC 575-002 Anasorb 747 rates at 760 mmHg and 25 °C
 1,2,3-trimethylbenzene 11.6 mL/min (2.78 L)
 1,2,4-trimethylbenzene 11.7 mL/min (2.81 L)
 1,3,5-trimethylbenzene 12.1 mL/min (2.90 L)

Reliable quantitation limits:

sampler	1,2,3-trimethylbenzene			1,2,4-trimethylbenzene			1,3,5-trimethylbenzene		
	RQL		SEE	RQL		SEE	RQL		SEE
	ppm	mg/m ³	(%)	ppm	mg/m ³	(%)	ppm	mg/m ³	(%)
226-01 Anasorb CSC	0.109	0.535	5.57	0.095	0.469	5.46	0.072	0.354	5.35
SKC 575-002 Anasorb 747*	0.775	3.81	9.26	0.729	3.58	9.26	0.656	3.22	9.34

*For diffusive samplers when sampling site atmospheric pressure and temperature are known. When either or both of these values are unknown, see Section 4.5 for applicable standard errors of estimate.

Special requirements: When using a 575-002 Anasorb 747 Passive Sampler, report the temperature and uncorrected sampling site atmospheric pressure. Refrigerated storage of collected samples is recommended but not required.

Status of method: Fully validated method. This method has been subjected to the established validation procedures of the Methods Development Team.

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1. General Discussion

For assistance with accessibility problems in using figures and illustrations presented in this method, please contact the Salt Lake Technical Center (SLTC) at (801) 233-4900. This procedure was designed and tested for internal use by OSHA personnel. Mention of any company name or commercial product does not constitute endorsement by OSHA.

1.1 Background

1.1.1 History

OSHA routinely samples for trimethylbenzene isomers and developed the partially validated method PV2091 in 1987¹. OSHA 1020 updates the status to fully validated, with changes to the flow rate, collection time, extraction solvent, and analytical conditions. Passive samplers were also added as a sampling option.

1.1.2 Toxic effects²

Animal Studies

1,3,5-Trimethylbenzene and 1,2,4-trimethylbenzene are both pharmacologically and toxicologically similar. The liquid is a primary skin irritant but systemic intoxication through skin exposure is unlikely. Acute exposure to vapor caused central nervous system depression in mice and death in rats due to respiratory arrest. Subchronic exposure to rats for 10-21 days resulted in no fatalities or other toxic effects. When this exposure is extended to 4 months, it caused reduced weight gain and central nervous system depression. Another observation was increased lymphopenia and neutrophilia, but this may have been caused by the benzene content in the solvent.

Human Studies

An investigation of 27 workers using a solvent mixture of 30% 1,3,5-trimethylbenzene and 50% 1,2,4-trimethylbenzene, for a number of years, showed a significant number of the individuals complaining of “nervousness, tension, anxiety, and asthmatic bronchitis.” There is not enough data to recommend a Skin, SEN, carcinogenicity, or TLV-STEL.

1.1.3 Workplace exposure³

All three trimethylbenzene isomers have multiple commercial uses and research applications. All three isomers are used as precursors for the manufacture of other chemicals. 1,3,5-Trimethylbenzene is used to make a phenol that is used as a noncoloring antioxidant and a thermal stabilizer for plastics, adhesives, rubber, and waxes. It can also be used to make mesitaldehyde which is a plant growth inhibitor. 1,2,3-Trimethylbenzene is used as a precursor to musk Tibetine. 1,2,4-Trimethylbenzene is mainly “used as a component in liquid scintillation cocktails for clinical analyses.” It is also used as a precursor to trimellitic acid and dehydrated to

¹ Gasser, F. Trimethylbenzenes (OSHA Method PV2091), 1987. United States Department of Labor, Occupational Safety & Health Administration Web site. <http://www.osha.gov/dts/sitc/methods/partial/pv2091/pv2091.html> (accessed January 2016).

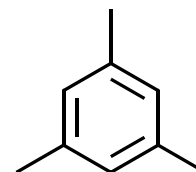
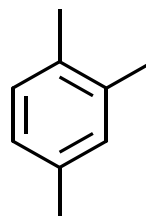
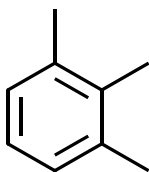
² American Conference of Governmental Industrial Hygienists, Inc. *Documentation of the Threshold Limit Values and Biological Exposure Indices*, 7th ed.; Cincinnati, OH, 2001; Vol. 2, Trimethyl benzene, isomers pp. 1-3.

³ Polymethylbenzenes, Published Online 4 Dec 2000. Kirk-Ortmer Encyclopedia of Chemical Technology Web site. <http://onlinelibrary.wiley.com/doi/10.1002/0471238961.1615122505011808.a01/pdf> (accessed February 2016).

trimellitic anhydride, which is then converted to PVC plasticizers, polyesters, water-soluble alkyd coatings, and polyamide-imide resins. National production volumes for 1,2,3-trimethylbenzene is 100,000 – 5000,000 lb/yr, 1,2,4-trimethylbenzene is 88,513,741 lb/yr, and 1,3,5-trimethylbenzene is 3,607,910 lb/yr.⁴

1.1.4 Physical properties and descriptive information⁵

analyte:	1,2,3-trimethylbenzene	1,2,4-trimethylbenzene	1,3,5-trimethylbenzene
synonyms:	hemimellitene	pseudocumene	mesitylene
solubility:	Practically insoluble in water. Miscible with alcohol, ether, benzene.		
IMIS number:	T205	T306	T407
CAS number:	526-73-8	95-63-6	108-67-8
molecular weight:	120.19	120.19	120.19
density:	0.8944	0.8761	0.8637
boiling point:	176 °C	168.9 °C	164.7 °C
melting point:	-25.37 °C	-43.78 °C	-44.8 °C
structural formula:	C ₉ H ₁₂	C ₉ H ₁₂	C ₉ H ₁₂
appearance:	liquid	liquid	liquid
odor:			peculiar
structural formula:			



Note: Mixed isomers IMIS is 2505 and CAS is 25551-13-7.

⁴ Chemical Data Access Tool (CDAT). United States Environmental Protection Agency Web site. http://java.epa.gov/oppt_chemical_search/ (accessed February 2016).

⁵ O'Neil, M.; Heckelman, P.; Koch, C.; Roman, K. *The Merck Index*; 14th ed.; Budavari, S., Ed.; Merck & Co. Inc.: Whitehouse Station, NJ, 2006; p 1020 and 1361.

This method was validated according to the OSHA SLTC “Validation Guidelines for Air Sampling Methods Utilizing Chromatographic Analysis”⁶. The Guidelines define analytical parameters, specify required laboratory tests, statistical calculations, and acceptance criteria. The analyte air concentrations throughout this method are based on the recommended sampling and analytical parameters. Air concentrations listed in ppm are referenced to 25 °C and 760 mmHg (101.3 kPa).

2. Sampling Procedure

All safety practices that apply to the work area being sampled should be followed. The sampling equipment should be attached to the worker in a manner that will not interfere with work performance or safety.

2.1 Apparatus

2.1.1 226-01 Anasorb CSC

Samples are collected with 6-mm x 70-mm glass sampling tubes packed with two sections of Anasorb CSC. The front section contains 100 mg and the back section contains 50 mg of Anasorb CSC 20/40 mesh. The sections are held in place and separated with foam and glass wool plugs. For this validation, commercially prepared tubes were purchased from SKC Inc. (catalog no. 226-01, lot no. 2000, batches 12397, 11907, 11986).

A sampling tube holder is required to protect the worker from the sharp end of the glass sampling tube.

Samples are collected using a personal sampling pump calibrated to within $\pm 5\%$ of the recommended flow rate with the sampling device in-line.

2.1.2 SKC 575-002 Anasorb 747

Samples are collected with a 575-002 Passive Sampler. These samplers contain 500 mg of Anasorb 747. For this validation, commercially available samplers were purchased from SKC Inc. (catalog no. P226200, lot no. 9219).

2.1.3 A thermometer and barometer are required to determine the sampling site air temperature and atmospheric pressure.

2.2 Reagents

None required

2.3 Technique

2.3.1 226-01 Anasorb CSC

Immediately before sampling, break off the ends of the flame-sealed tube to provide an opening approximately half the internal diameter of the tube. Wear eye protection

⁶ Eide, M.; Simmons, M.; Hendricks, W. Validation Guidelines for Air Sampling Methods Utilizing Chromatographic Analysis, 2010. United States Department of Labor, Occupational Safety & Health Administration Web site. <http://www.osha.gov/dts/sltc/methods/chromguide/chromguide.pdf> (accessed March 2016).

when breaking ends. Use sampling tube holders to minimize the hazard to the worker from the broken ends of the tubes. All tubes should be from the same lot.

The smaller section of adsorbent in the adsorbent tube is used as a back-up and is positioned nearest the sampling pump. Attach the tube holder (with the adsorbent tube) to the sampling pump so that the adsorbent tube is in an approximately vertical position with the inlet facing down in the worker's breathing zone during sampling. Position the sampling pump, tube holder, and tubing so they do not impede work performance or safety.

Draw air directly into the inlet of the sampling tube holder. The air being sampled should not pass through any hose or tubing before entering the sampling tube.

Sample for up to 240 min at 50 mL/min (12 L) when using Anasorb CSC, Coconut Charcoal to collect TWA (long-term) samples.

After sampling for the appropriate time, remove the adsorbent tube and seal it with plastic end caps. Seal each sample end-to-end with a Form OSHA-21 as soon as possible.

Submit at least one blank sample with each set of samples. Handle the blank sample in the same manner as the other samples except draw no air through it.

Record sample air volume (liters), sampling time (min) and sampling rate (mL/min) for each sample, along with any potential interference on the Form OSHA-91A.

Submit the samples to the laboratory for analysis as soon as possible after sampling. If delay is unavoidable, store the samples in a refrigerator as a precaution.

Ship any bulk sample(s) separate from the air samples.

2.3.2 SKC 575-002 Anasorb 747 Passive Samplers (In general, follow the manufacturer's instructions.)

Remove the sampler from its air-tight package.

Record the start time on the Form OSHA-91A. Remove the cover when ready to begin sampling. CAUTION - The sampler immediately begins to sample when the cover is removed. Keep the press-on cover in the package for later use.

Attach the sampler to the worker near his/her breathing zone with the perforations in the sampler facing forward. Assure that the area directly in front of the sampler is unobstructed throughout the sampling period.

Sample for up to 240 min when using SKC 575-002 to collect TWA (long-term) samples. The sampling rates for 1,2,3-trimethylbenzene, 1,2,4-trimethylbenzene, and 1,3,5-trimethylbenzene using SKC 575-002 are 11.6, 11.7, and 12.1 mL/min at 760 mmHg and 25 °C.

Sample for 15 min when using SKC 575-002 to collect short-term samples. The sampling rates for 1,2,3-trimethylbenzene, 1,2,4-trimethylbenzene, and 1,3,5-trimethylbenzene using SKC 575-002 are 11.6, 11.7, and 12.1 mL/min at 760 mmHg and 25 °C.

At the end of the sampling period, immediately detach the sampler from the worker and reattach the cover onto the front of the sampler to cover the perforations. Record the

stop time on the Form OSHA-91A. Return each sampler to its package, close the package, and seal it with a Form OSHA-21.

Prepare a blank in a low background area by removing an unused sampler from its package. Then remove the cover. Immediately replace the cover onto the sampler. Return the blank sampler to its package, close the package, and seal it with a Form OSHA-21.

Verify that the sampling times are properly recorded on the Form OSHA-91A for each sample. Also, identify blank samples on this form.

Record the ambient temperature and atmospheric pressure at the sampling site on the Form OSHA-91A.

List any compounds that could be considered potential interferences, especially solvents that are being used in the sampling area.

Submit the samplers to the laboratory for analysis as soon as possible. If delay is unavoidable, store the samples in a refrigerator as a precaution.

Ship any bulk sample(s) in a container separate from the air samples.

3. Analytical Procedure

3.1 Apparatus

Gas chromatograph equipped with an FID. An Agilent 7890 Series GC System was used in this validation.

GC injection port liner. Restek Sky 4.0-mm ID Low Pressure Drop Precision Inlet Liner w/wool (Restek catalog no. 23309.1, or equivalent) was used in this validation.

GC column capable of separating the isomers of trimethylbenzene from each other, the extraction solvent, potential interferences, and internal standard. An Agilent J&W DB-624 20-m × 0.18-mm i.d. (film thickness 1.0- μ m) capillary column was used in this validation.

Electronic integrator or other suitable means of measuring GC detector response. A Waters Empower 3 Data System was used in this validation.

Glass vials (clear) with PTFE-lined crimp caps. In this validation 2.0-mL vials were used.

Glass vials (amber) with PTFE-lined screw caps. In this validation 4.0-mL vials were used.

A dispenser capable of delivering 1.0 mL of extraction solvent to prepare standards and samples. If a dispenser is not available, 1.0-mL volumetric pipettes can be used.

Class A volumetric flasks of convenient sizes for standard preparation. In this validation 1-mL, 2-mL, and 5-mL flasks were used.

Calibrated microliter syringes of convenient sizes for standard preparation. An SGE 25- μ L syringe was used in this validation.

Mechanical shaker. An Eberbach Model 6000 shaker was used in this validation.

Mechanical rotator. A Thermo Scientific Labquake Shaker Rotisserie Model 415110 was used in this validation.

3.2 Reagents

Carbon disulfide (CS₂), 75-15-0, reagent grade or better. The CS₂ used in this validation was low benzene grade 99.9% (lot nos. 144325 and 143607) purchased from Fisher (Phillipsburg, NJ).

N,N-Dimethylformamide (DMF), 68-12-2, reagent grade or better. The DMF used in this validation was 99% (lot no. 1353807) purchased from Acros (Fair View, NJ).

n-Hexylbenzene, 1077-16-3, reagent grade or better. The *n*-hexylbenzene used in this validation was 97% (lot no. A0238811) purchased from Acros (NJ).

1,2,3-Trimethylbenzene, 526-73-8, tech grade or better. The 1,2,3-trimethylbenzene used in this validation was 90% (lot no. MKBR7837V) purchased from Aldrich (St. Louis, MO) and 80% (lot no. D44DOPM) purchased from TCI (Portland, OR). Find the highest purity possible and screen the isomer to ensure that it doesn't add a positive bias to the other isomers.

1,2,4-Trimethylbenzene, 95-63-6, reagent grade or better. The 1,2,4-trimethylbenzene used in this validation was 98% (lot no. MKBK8538V) purchased from Aldrich (St. Louis, MO).

1,3,5-Trimethylbenzene, 108-67-8, reagent grade or better. The 1,3,5-trimethylbenzene used in this validation was 98% (lot nos. BCBN2251V and BCBL3127V) purchased from Sigma Aldrich (St. Louis, MO).

Extraction solvent. The extraction solvent used in this validation consisted of 0.25 µL/mL *n*-hexylbenzene in 99/1 (v/v) CS₂/DMF. The *n*-hexylbenzene was added as an internal standard. The extraction efficiency is affected by the extraction solvent, the internal standard, the sampling medium, and the technique used to extract the samples. Other reagents and techniques than described in this method can be used provided they are tested as specified in the validation guidelines.⁷

3.3 Standard preparation

Prepare a concentrated stock solution of a 1:1:1 volume mixture of 1,2,3-trimethylbenzene, 1,2,4-trimethylbenzene, and 1,3,5-trimethylbenzene. Prepare working analytical standards by injecting microliter amounts of the concentrated stock standard into 2-mL volumetric flasks containing 1 mL of the extraction solvent and then fill to the mark on the flask with extraction solvent. For example, to prepare a target level standard based on active sampling at the recommended flowrate and time, inject 10 µL of a stock solution containing 268.20, 286.16, 282.24 mg/mL of 1,2,3-trimethylbenzene, 1,2,4-trimethylbenzene, and 1,3,5-trimethylbenzene into 2 mL of extraction solvent.

Bracket sample concentrations with standard concentrations. If upon analysis, sample concentrations fall outside the range of prepared standards, prepare and analyze additional standards to confirm instrument response, or dilute high samples with extraction solvent and reanalyze the diluted samples.

⁷ Eide, M.; Simmons, M.; Hendricks, W. Validation Guidelines for Air Sampling Methods Utilizing Chromatographic Analysis, 2010. United States Department of Labor, Occupational Safety & Health Administration Web site. <http://www.osha.gov/dts/sltc/methods/chromguide/chromguide.pdf> (accessed March 2016).

3.4 Sample preparation

3.4.1 226-01 Anasorb CSC

Remove the plastic end caps from the sample tube and carefully transfer each section of the adsorbent to separate 2-mL vials. Discard the glass tube, foam, and glass wool plugs.

Add 1.0 mL of extraction solution to each vial and immediately seal the vials with PTFE-lined caps.

Extract the samples for 30 min by shaking.

3.4.2 SKC 575-002 Anasorb 747 (In general, follow the manufacturer's instructions.)

Remove the plug and foam insert. Carefully transfer media to a 4-mL amber screw-cap vial.

Slowly add 2.0 mL of extraction solvent and cap the vial.

Mount the vials on the rotator rack and rotate the samplers for 30 min.

Transfer the solvent extract to a 2-mL vial.

3.5 Analysis

3.5.1 Analytical conditions

GC conditions

oven temperature:	40 °C (hold 2.25 min), ramp to 90 °C at 7 °C/min (hold 0 min), ramp to 225 °C at 25 °C/min (hold 0.2 min)
injector temperature:	250 °C
run time:	15 min
column:	Agilent J&W DB-624 capillary column, 20-m × 0.18-mm i.d., d _f = 1.0-µm, or equivalent
column mode:	constant pressure (18 psi)
initial column gas flow:	1.5 mL/min (hydrogen)
septum purge:	3.0 mL/min (hydrogen)
injection size:	1.0 µL (150 to 1 split)
inlet liner:	Restek Sky 4.0-mm ID Low Pressure Drop Precision Inlet Liner w/wool (Restek catalog no. 23309.1, or equivalent)
retention times:	1.7 min (CS ₂) 8.6 min (DMF) 11.3 min (1,3,5-trimethylbenzene) 11.7 min (1,2,4-trimethylbenzene) 12.0 min (1,2,3-trimethylbenzene) 14.1 min (n-hexylbenzene ISTD)

FID conditions

detector temperature:	250 °C
hydrogen flow:	30 mL/min
air flow:	400 mL/min
nitrogen make up flow:	25 mL/min

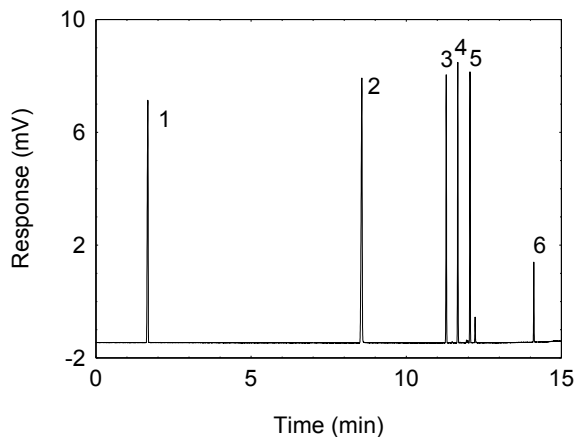


Figure 3.5.1. Chromatogram obtained at the target concentration with the recommended analytical conditions (1: CS₂; 2: DMF; 3: 1,3,5-trimethylbenzene; 4: 1,2,4-trimethylbenzene; 5: 1,2,3-trimethylbenzene; 6: n-hexylbenzene (ISTD)).

3.5.2 An internal standard (ISTD) calibration method is used. A linear calibration curve can be constructed by plotting ISTD-corrected response of standard injections versus micrograms of analyte per sample. When adding reporting limit standards to the calibration, the curves can be weighted to reduce bias at the reporting limit. Bracket the samples with freshly prepared analytical standards over a range of concentrations.

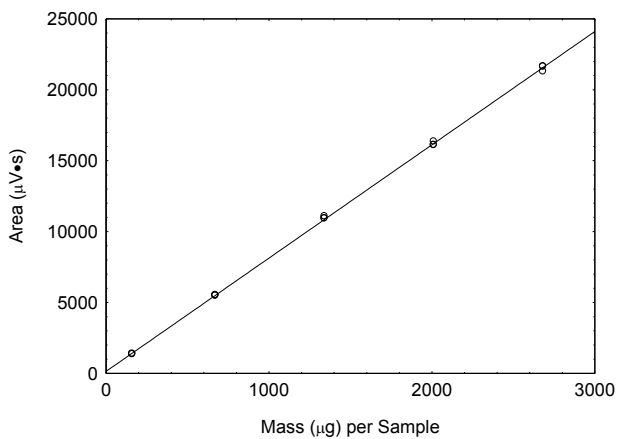


Figure 3.5.2.1. Calibration curve for 1,2,3-trimethylbenzene ($y = 7.99x + 145$).

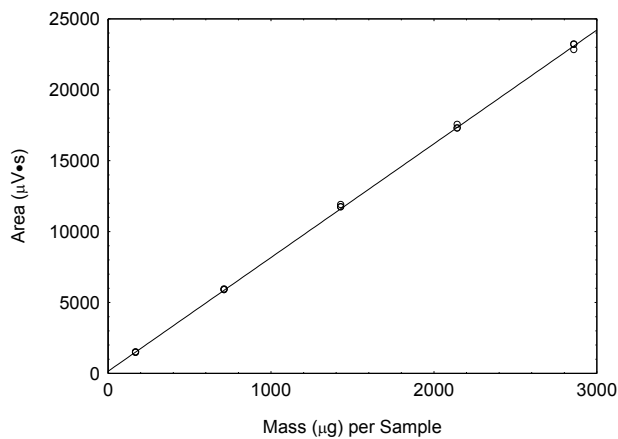


Figure 3.5.2.2. Calibration curve for 1,2,4-trimethylbenzene ($y = 8.02x + 153$).

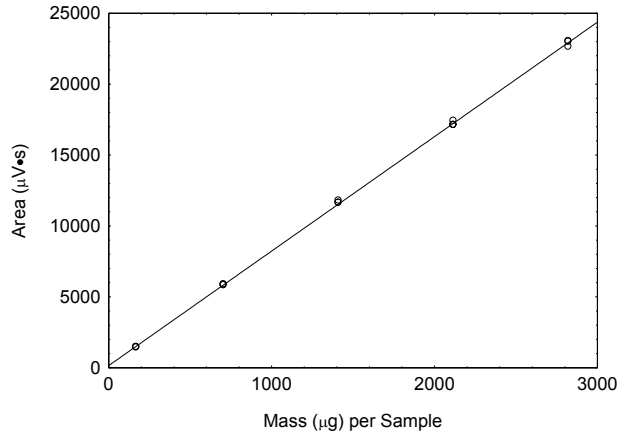


Figure 3.5.2.3. Calibration curve for 1,3,5-trimethylbenzene ($y = 8.07x + 153$).

3.6 Interferences (analytical)

- 3.6.1 Any compound that produces an FID response and has a similar retention time as the analyte or internal standard is a potential interference. If any potential interferences are reported, they should be considered before samples are extracted. Generally, chromatographic conditions can be altered to separate interferences from the analyte.
- 3.6.2 When necessary, the identity of an analyte peak can be confirmed with additional analytical data or procedures (Section 4.10).

3.7 Calculations

3.7.1 226-01 Anasorb CSC

The amount of trimethylbenzene isomers per sample is obtained from calculating the micrograms per sample (M) for each isomer and then summing the results. The back section is analyzed primarily to determine the extent of sampler saturation. If any analyte is found on the back section, it is added to the amount on the front section. If more than 20% of the total amount is found on the back section, report that the sampler may have been saturated on the Form OSHA-91B. This total amount is then corrected by subtracting the total amount (if any) found on the blank. The air concentration is calculated using the following formulas.

$$M = \frac{M_{obs} - M_{blk}}{E_E}$$

where M is micrograms per sample blank corrected and extraction corrected
 M_{obs} is micrograms per sample
 M_{blk} is micrograms per sample blank
 E_E is extraction efficiency, in decimal form

$$C_M = \frac{M_{sum}}{V}$$

where C_M is concn by weight in air (mg/m^3)
 M_{sum} is summation of micrograms per sample for each isomer blank corrected and extraction corrected
 V is liters of air sampled

$$C_V = \frac{C_M V_M}{M_r}$$

where C_V is concn by volume (ppm)
 C_M is concn by weight in air (mg/m^3)
 V_M is 24.46 (molar volume at NTP)
 M_r is molecular weight 120.19

3.7.2 SKC 575-002 Anasorb 747

The mass (M) of trimethylbenzene isomers is calculated as in section 3.7.1. The air concentration is calculated using the following formulas.

$$R_{SS} = R_{NTP} \left(\frac{T_{SS}}{T_{NTP}} \right)^{\frac{3}{2}} \left(\frac{P_{NTP}}{P_{SS}} \right)$$

where R_{SS} is the sampling rate at the sampling site (mL/min)
 R_{NTP} is the sampling rate at NTP (mL/min)
 T_{SS} is the temp at the sampling site (K)
 T_{NTP} is 298.2 K
 P_{SS} is the sampling site pressure (mmHg)
 P_{NTP} is 760 mmHg

$$C_M = \frac{M_{sum} 1000}{R_{SS} t}$$

where C_M is concn by weight in air (mg/m³)
 M_{sum} is summation of micrograms per sample for each isomer blank corrected and extraction corrected
 R_{SS} is the sampling rate at the sampling site (mL/min)
 t is the sampling time (min)

$$C_V = \frac{C_M V_M}{M_r}$$

where C_V is concentration by volume (ppm)
 V_M is 24.46 (molar volume at NTP)
 C_M is concentration by weight (mg/m³)
 M_r is molecular weight 120.19

If the sampling site temperature is not provided, assume that it is 22.2 °C. If the sampling site atmospheric pressure is not given, calculate an approximate value based on the sampling site elevation from the following equation.

$$P_{SS} = AE^2 - BE + 760$$

P_{SS} is the approximate atmospheric pressure (mmHg)
 E is the sampling site elevation (ft)
 A is 3.768×10^{-7} mmHg/ft²
 B is 0.02741 mmHg/ft

4. Method Validation

General instruction for the laboratory validation of OSHA sampling and analytical methods that employ chromatographic analysis is presented in "Validation Guidelines for Air Sampling Methods Utilizing Chromatographic Analysis"⁸. These Guidelines detail required validation tests, show examples of statistical calculations, list validation acceptance criteria, and define analytical parameters. Air concentrations listed in ppm are referenced to 25 °C and 760 mmHg (101.3 kPa).

4.1 Detection limit of the analytical procedure (DLAP)

The DLAP is measured as the mass of analyte introduced onto the chromatographic column. Ten analytical standards were prepared separately with equally descending increments of 1,2,3-trimethylbenzene, 1,2,4-trimethylbenzene, and 1,3,5-trimethylbenzene with the highest standard containing 11.0, 10.6, and 10.6 µg/mL each. This is the concentration that would

⁸ Eide, M.; Simmons, M.; Hendricks, W. Validation Guidelines for Air Sampling Methods Utilizing Chromatographic Analysis, 2010. United States Department of Labor, Occupational Safety & Health Administration Web site. <http://www.osha.gov/dts/sltc/methods/chromguide/chromguide.pdf> (accessed March 2016).

produce a peak approximately 10 times the response of a reagent blank at or near the chromatographic retention time of the analyte. These standards and the reagent blank were analyzed with the recommended analytical parameters (1- μ L injection with a 150:1 split). The data obtained were used to determine the required parameters (standard error of estimate and slope) for the calculation of the DLAP. Values for 1,2,3-trimethylbenzene of 1.15 and 4.59 were obtained for the slope and standard error of estimate respectively. The DLAP was calculated to be 11.9 pg. Values for 1,2,4-trimethylbenzene of 1.17 and 4.69 were obtained for the slope and standard error of estimate respectively. The DLAP was calculated to be 12.0 pg. Values for 1,3,5-trimethylbenzene of 1.28 and 5.42 were obtained for the slope and standard error of estimate respectively. The DLAP was calculated to be 12.7 pg.

Table 4.1.1
Detection Limit of the Analytical Procedure
for 1,2,3-Trimethylbenzene

concentration (μ g/mL)	mass on column (pg)	area counts (μ V \cdot s)
0	0	0
0.894	5.96	14.8
2.01	13.4	26.6
3.13	20.9	30.9
4.25	28.3	39.5
5.36	35.7	48.9
6.48	43.2	51.8
7.60	50.7	65.7
8.72	58.1	66.8
9.83	65.5	77.4
11.0	73.3	97.3

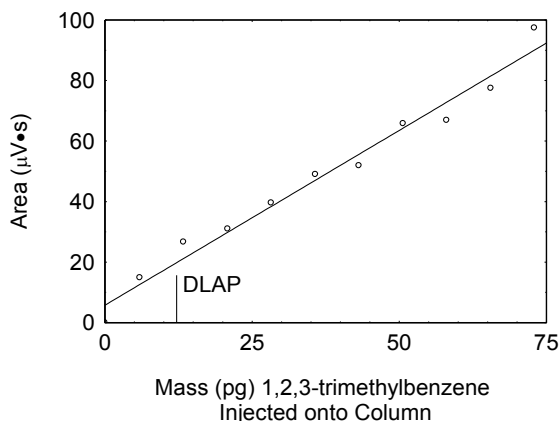


Figure 4.1.1. Plot of data to determine the DLAP for 1,2,3-trimethylbenzene ($y = 1.15x + 5.80$).

Table 4.1.2
Detection Limit of the Analytical Procedure
for 1,2,4-Trimethylbenzene

concentration (μ g/mL)	mass on column (pg)	area counts (μ V \cdot s)
0	0	19.9
0.864	5.76	20.7
1.94	12.9	24.1
3.02	20.1	36.0
4.10	27.3	41.5
5.18	34.5	52.3
6.26	41.7	57.9
7.34	48.9	69.7
8.42	56.1	71.4
9.50	63.3	90.8
10.6	70.7	102

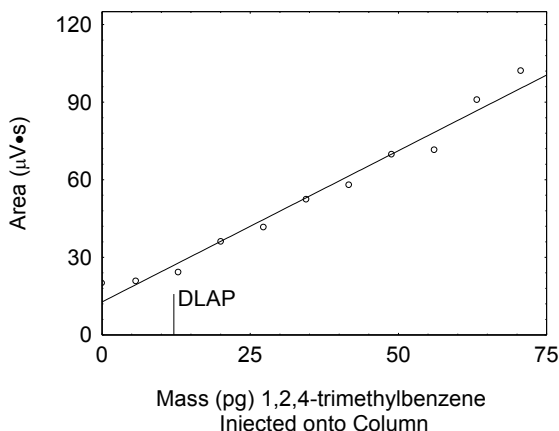


Figure 4.1.2. Plot of data to determine the DLAP for 1,2,4-trimethylbenzene ($y = 1.17x + 12.7$).

Table 4.1.3
Detection Limit of the Analytical Procedure
for 1,3,5-Trimethylbenzene

concentration ($\mu\text{g/mL}$)	mass on column (μg)	area counts ($\mu\text{V}\bullet\text{s}$)
0	0	0
0.864	5.76	10.9
1.94	12.9	25.8
3.02	20.1	33.2
4.10	27.3	37.5
5.18	34.5	46.7
6.26	41.7	71.6
7.34	48.9	65.6
8.42	56.1	73.2
9.50	63.3	85.6
10.6	70.7	92.6

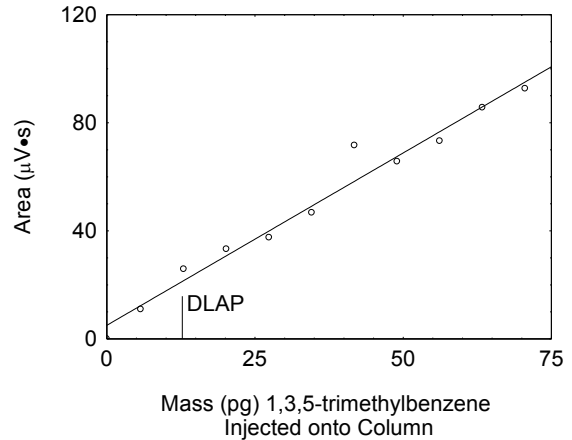


Figure 4.1.3. Plot of data to determine the DLAP for 1,3,5-trimethylbenzene ($y = 1.28x + 5.12$).

4.2 Detection limit of the overall procedure (DLOP) and reliable quantitation limit

226-01 Anasorb CSC

The DLOP is measured as mass per sample and expressed as equivalent air concentrations based on the recommended sampling parameters. Ten 226-01 Anasorb CSC samplers were spiked with equally descending increments of analyte, such that the highest sampler loading for 1,2,3-trimethylbenzene, 1,2,4-trimethylbenzene and 1,3,5-trimethylbenzene were 11.8, 12.6, and 12.4 $\mu\text{g/sample}$ each. This is the amount spiked on a sampler that would produce a peak approximately 10 times the response of a sample blank at or near the chromatographic retention time of the analyte. These spiked samplers and the sample blank were analyzed with the recommended analytical parameters, and the data obtained used to calculate the required parameters (standard error of estimate and the slope) for the calculation of the DLOP. Values for 1,2,3-trimethylbenzene of 7.65 and 4.91 were obtained for the slope and standard error of estimate respectively. The DLOP was calculated to be 1.93 $\mu\text{g/sample}$ (0.033 ppm or 0.161 mg/m^3). Values for 1,2,4-trimethylbenzene of 8.08 and 4.55 were obtained for the slope and standard error of estimate respectively. The DLOP was calculated to be 1.69 $\mu\text{g/sample}$ (0.029 ppm or 0.141 mg/m^3). Values for 1,3,5-trimethylbenzene of 8.59 and 3.65 were obtained for the slope and standard error of estimate respectively. The DLOP was calculated to be 1.27 $\mu\text{g/sample}$ (0.022 ppm or 0.106 mg/m^3).

Table 4.2.1
Detection Limit of the Overall Procedure
for 1,2,3-Trimethylbenzene on 226-01 Anasorb CSC

mass per sample (μg)	area counts ($\mu\text{V}\bullet\text{s}$)
0	0
0.966	11.9
2.17	21.5
3.38	37.2
4.59	45.1
5.79	50.6
7.00	68.4
8.21	69.7
9.41	72.2
10.6	82.6
11.8	96.6

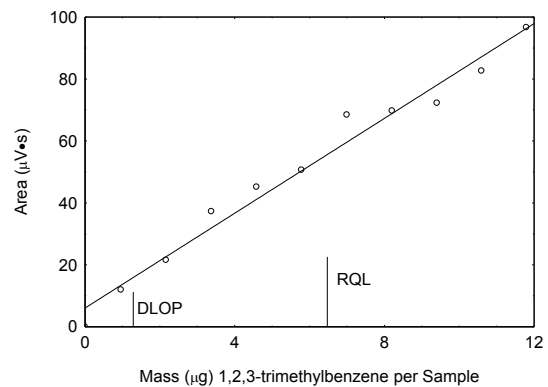


Figure 4.2.1. Plot of data to determine the DLOP for 1,2,3-trimethylbenzene ($y = 7.65x + 6.06$).

Table 4.2.2
Detection Limit of the Overall Procedure
for 1,2,4-Trimethylbenzene on 226-01 Anasorb CSC

mass per sample (μg)	area counts ($\mu\text{V}\cdot\text{s}$)
0	0
1.03	16.4
2.32	31.4
3.61	34.3
4.89	47.7
6.18	65.7
7.47	70.4
8.76	76.3
10.0	84.6
11.3	96.7
12.6	109

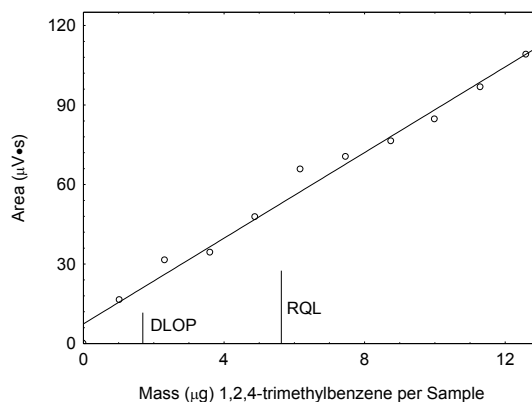


Figure 4.2.2. Plot of data to determine the DLOP for 1,2,4-trimethylbenzene ($y = 8.08x + 7.43$).

Table 4.2.3
Detection Limit of the Overall Procedure
for 1,3,5-Trimethylbenzene on 226-01 Anasorb CSC

mass per sample (μg)	area counts ($\mu\text{V}\cdot\text{s}$)
0	0
1.02	13.9
2.29	27.7
3.56	32.4
4.83	47.5
6.10	55.8
7.37	69.1
8.64	77.0
9.91	95.6
11.2	101
12.4	105

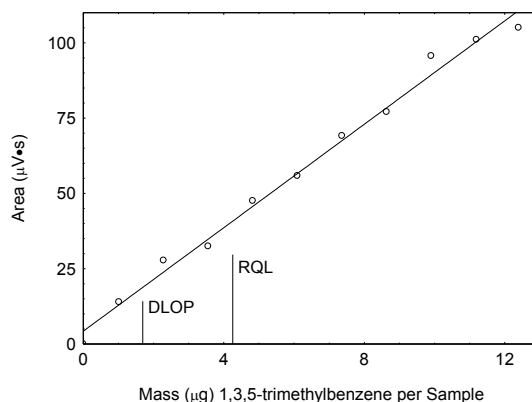


Figure 4.2.3. Plot of data to determine the DLOP for 1,3,5-trimethylbenzene ($y = 8.59x + 4.27$).

The RQL is considered the lower limit for precise quantitative measurements. It is determined from the regression line parameters that were obtained for the calculation of DLOP providing 75% to 125% of the analyte is recovered. The RQLs for 1,2,3-trimethylbenzene, 1,2,4-trimethylbenzene, and 1,3,5-trimethylbenzene are $6.42 \mu\text{g}/\text{sample}$ (0.109 ppm or $0.535 \text{ mg}/\text{m}^3$), $5.63 \mu\text{g}/\text{sample}$ (0.095 ppm or $0.469 \text{ mg}/\text{m}^3$), and $4.25 \mu\text{g}/\text{sample}$ (0.072 ppm or $0.354 \text{ mg}/\text{m}^3$). Recoveries at these concentrations are 116%, 117%, and 104%.

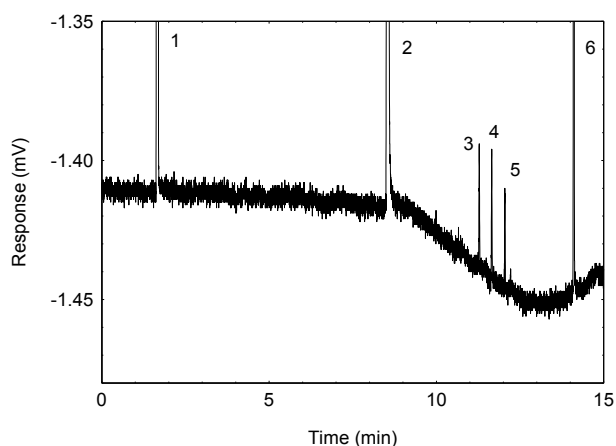


Figure 4.2.4. Chromatogram of the RQL (1:CS₂; 2: DMF; 3: 1,3,5-trimethylbenzene; 4: 1,2,4-trimethylbenzene; 5: 1,2,3-trimethylbenzene; 6: n-hexylbenzene (ISTD)).

SKC 575-002 Anasorb 747

The DLOP is measured as mass per sample and expressed as equivalent air concentrations based on the recommended sampling parameters. Ten SKC 575-002 Anasorb 747 passive samples were spiked with equally descending increments of analyte, such that the highest sampler loading for 1,2,3-trimethylbenzene, 1,2,4-trimethylbenzene and 1,3,5-trimethylbenzene were 23.7, 25.2, and 24.9 µg/sample each. This is the amount spiked on a sampler that would produce a peak approximately 10 times the response of a sample blank at or near the chromatographic retention time of the analyte. These spiked samplers and the sample blank were analyzed with the recommended analytical parameters, and the data obtained used to calculate the required parameters (standard error of estimate and the slope) for the calculation of the DLOP. Values for 1,2,3-trimethylbenzene of 4.17 and 4.41 were obtained for the slope and standard error of estimate respectively. The DLOP was calculated to be 3.17 µg/sample (0.232 ppm or 1.14 mg/m³). Values for 1,2,4-trimethylbenzene of 4.23 and 4.26 were obtained for the slope and standard error of estimate respectively. The DLOP was calculated to be 3.02 µg/sample (0.219 ppm or 1.08 mg/m³). Values for 1,3,5-trimethylbenzene of 4.70 and 4.39 were obtained for the slope and standard error of estimate respectively. The DLOP was calculated to be 2.80 µg/sample (0.196 ppm or 0.966 mg/m³).

Table 4.2.4
Detection Limit of the Overall Procedure
for 1,2,3-Trimethylbenzene
on SKC 575-002 Anasorb 747

mass per sample (µg)	area counts (µV•s)
0	0
1.93	0
4.34	22.6
6.76	29.7
9.17	39.9
11.6	47.6
14.0	62.5
16.4	74.3
18.8	76.7
21.2	83.0
23.7	98.4

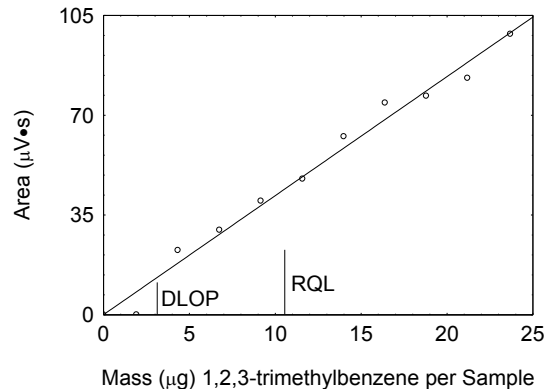


Figure 4.2.5. Plot of data to determine the DLOP for 1,2,3-trimethylbenzene ($y = 4.17x + 0.073$).

Table 4.2.5
Detection Limit of the Overall Procedure
for 1,2,4-Trimethylbenzene
on SKC 575-002 Anasorb 747

mass per sample (µg)	area counts (µV•s)
0	0
2.06	17.5
4.64	18.4
7.21	36.6
9.79	42.0
12.4	55.1
14.9	66.8
17.5	81.2
20.1	84.4
22.7	105
25.2	105

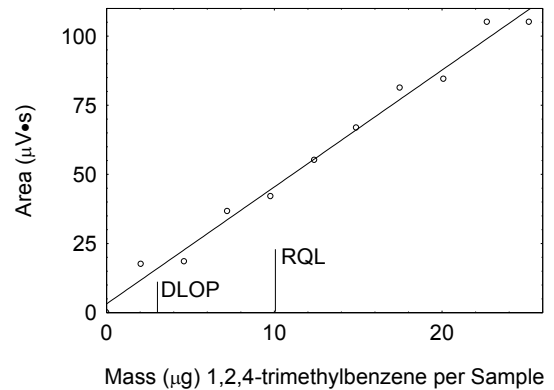


Figure 4.2.6. Plot of data to determine the DLOP for 1,2,4-trimethylbenzene ($y = 4.23x + 3.17$).

Table 4.2.6
Detection Limit of the Overall Procedure
for 1,3,5-Trimethylbenzene
on SKC 575-002 Anasorb 747

mass per sample (μg)	area counts ($\mu\text{V}\cdot\text{s}$)
0	0
2.03	0
4.57	24.5
7.11	32.7
9.65	44.8
12.2	61.0
14.7	69.3
17.3	82.8
19.8	94.3
22.4	97.3
24.9	116

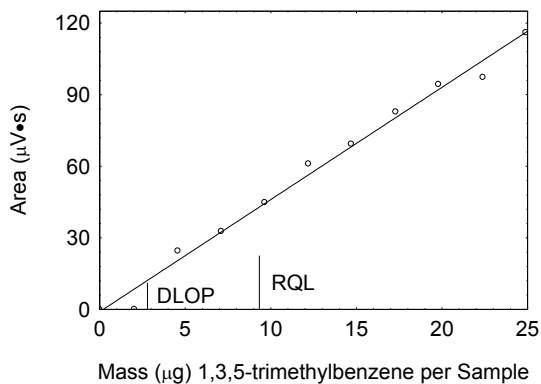


Figure 4.2.7. Plot of data to determine the DLOP for 1,3,5-trimethylbenzene ($y = 4.70x - 0.972$).

The RQL is considered the lower limit for precise quantitative measurements. It is determined from the regression line parameters that were obtained for the calculation of DLOP providing 75% to 125% of the analyte is recovered. The RQLs for 1,2,3-trimethylbenzene, 1,2,4-trimethylbenzene, and 1,3,5-trimethylbenzene are $10.6 \mu\text{g}/\text{sample}$ (0.775 ppm or $3.81 \text{ mg}/\text{m}^3$), $10.1 \mu\text{g}/\text{sample}$ (0.729 ppm or $3.58 \text{ mg}/\text{m}^3$), and $9.34 \mu\text{g}/\text{sample}$ (0.656 ppm or $3.22 \text{ mg}/\text{m}^3$). Recoveries at these concentrations are 98.3%, 93.8%, and 101%.

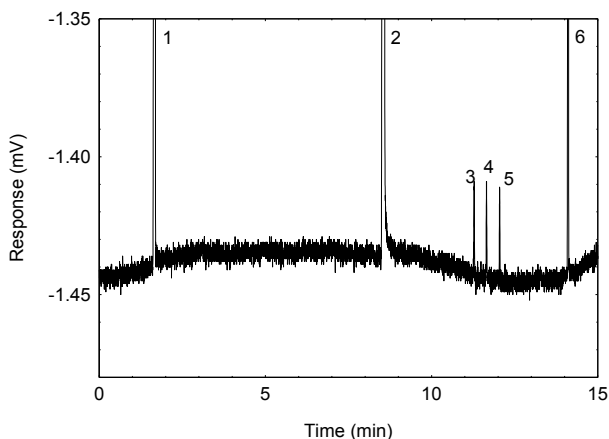


Figure 4.2.8. Chromatogram of the RQL (1:CS₂; 2: DMF; 3: 1,3,5-trimethylbenzene; 4: 1,2,4-trimethylbenzene; 5: 1,2,3-trimethylbenzene; 6: n-hexylbenzene (ISTD)).

4.3 Precision of the analytical method

The precision of the analytical method measured as the mass equivalent to the standard error of estimate determined from the linear regression of data points from standards over a range that covers 0.1 to 2 times the target concentration for the sampler with the highest mass loading. Calibration curves were constructed and shown in Section 3.5.2 from the three injections of five standards. The standard error of estimate for 1,2,3-trimethylbenzene, 1,2,4-trimethylbenzene, and 1,3,5-trimethylbenzene are -2.30, -2.16, and -1.86 μg each.

Table 4.3.1
Instrument Calibration 1,2,3-Trimethylbenzene

\times target concn ($\mu\text{g}/\text{sample}$)	0.1 \times	0.5 \times	1.0 \times	1.5 \times	2.0 \times
area counts	1362	5481	10940	16100	21290
($\mu\text{V}\cdot\text{s}$)	1392	5535	11080	16120	21660
	1353	5489	10930	16360	21630

Table 4.3.2
Instrument Calibration 1,2,4-Trimethylbenzene

× target concn (µg/sample)	0.1×	0.5×	1.0×	1.5×	2.0×
area counts	1456	5874	11720	17260	22800
(µV·s)	1490	5923	11870	17270	23200
	1444	5873	11700	17520	23170

Table 4.3.3
Instrument Calibration 1,3,5-Trimethylbenzene

× target concn (µg/sample)	0.1×	0.5×	1.0×	1.5×	2.0×
area counts	1445	5826	11630	17130	22640
(µV·s)	1480	5890	11790	17130	23040
	1432	5833	11640	17410	22990

4.4 Storage stability test

4.4.1 226-01 Anasorb CSC

Storage samples for 1,2,3-trimethylbenzene, 1,2,4-trimethylbenzene, and 1,3,5-trimethylbenzene were prepared by sampling a dynamically generated controlled test atmosphere using the recommended sampling parameters. The concentrations for ambient storage of 1,2,3-trimethylbenzene, 1,2,4-trimethylbenzene, and 1,3,5-trimethylbenzene in the test atmosphere were the target concentration (23.7, 25.3, 25.0 ppm, or 116.6, 124.4, 122.7 mg/m³) and the relative humidity was 65.8% at 24.7 °C. The concentrations for refrigerated storage of 1,2,3-trimethylbenzene, 1,2,4-trimethylbenzene, and 1,3,5-trimethylbenzene in the test atmosphere were the target concentration (23.8, 25.4, 25.1 ppm, or 117.1, 124.9, 123.2 mg/m³) and the relative humidity was 64.7% at 26.1 °C. Eighteen storage samples were prepared for each test. Three samples were analyzed on the day of generation. Fifteen samples were stored at reduced temperature (4 °C) and the other fifteen were stored in a closed drawer at ambient temperature (about 22 °C). At 3-4 day intervals, three samples were selected from each of the two storage sets and analyzed. Sample results are not corrected for extraction efficiency.

Table 4.4.1.1
Storage Test for 1,2,3-Trimethylbenzene
on 226-01 Anasorb CSC

Time (days)	ambient storage			refrigerated storage		
	recovery (%)			recovery (%)		
0	88.43	92.56	92.05	90.29	94.59	92.20
3	94.10	91.84	89.10	96.37	94.63	93.24
7	95.57	92.62	93.77			
8				95.16	93.61	90.97
10	93.09	92.55	87.52	90.96	91.03	90.66
14				86.38	84.86	87.36
15	94.97	91.64	91.74			
18	84.73	85.67	88.63			
19				87.89	91.18	87.47

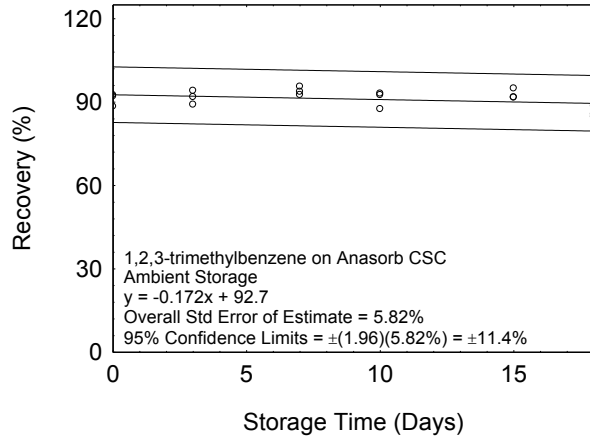


Figure 4.4.1.1. Ambient storage test for 1,2,3-trimethylbenzene on 226-01 Anasorb CSC.

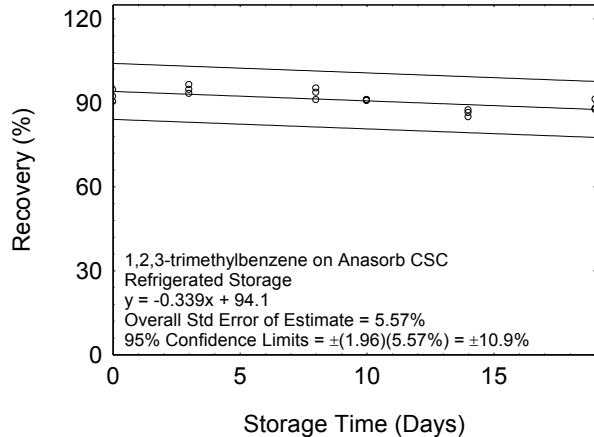


Figure 4.4.1.2. Refrigerated storage test for 1,2,3-trimethylbenzene on 226-01 Anasorb CSC.

Table 4.4.1.2
Storage Test for 1,2,4-Trimethylbenzene
on 226-01 Anasorb CSC

time (days)	ambient storage recovery (%)			refrigerated storage recovery (%)		
	0	91.84	96.04	95.37	93.56	97.95
3	97.51	95.25	92.30	99.74	98.07	96.38
7	98.95	96.26	97.22			
8				98.67	97.26	94.55
10	96.53	96.04	92.34	94.89	95.07	94.56
14				91.62	89.78	92.37
15	98.54	95.06	95.21			
18	88.99	89.77	92.71			
19				92.60	95.20	91.24

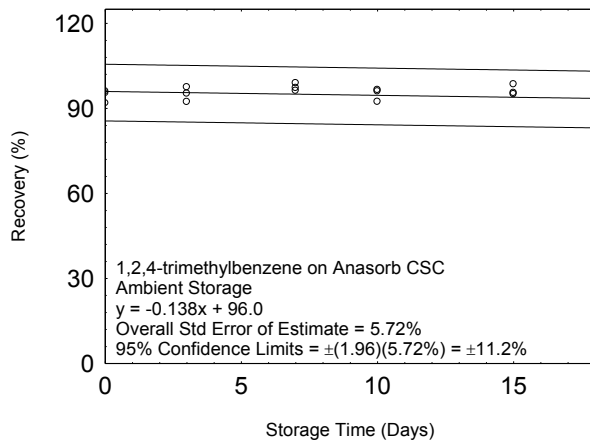


Figure 4.4.1.3. Ambient storage test for 1,2,4-trimethylbenzene on 226-01 Anasorb CSC.

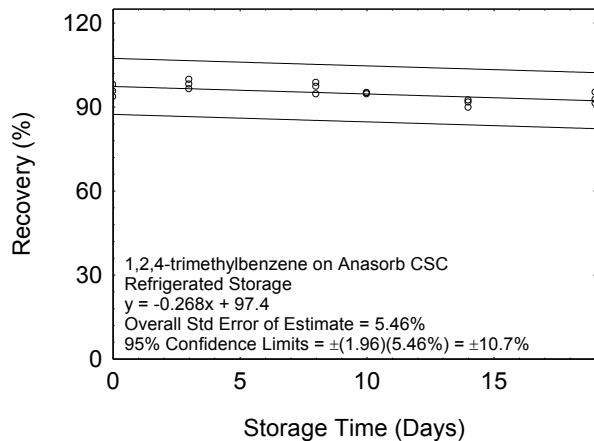


Figure 4.4.1.4. Refrigerated storage test for 1,2,4-trimethylbenzene on 226-01 Anasorb CSC.

Table 4.4.1.3
Storage Test for 1,3,5-Trimethylbenzene
on 226-01 Anasorb CSC

time (days)	ambient storage recovery (%)			refrigerated storage recovery (%)		
	0	94.91	99.10	98.22	96.46	100.9
3	97.51	95.25	92.30	102.9	101.0	99.37
7	102.0	99.58	100.2			
8				101.8	100.3	97.55
10	99.61	98.97	97.91	98.25	98.40	98.01
14				97.06	95.34	97.27
15	101.9	98.04	98.41			
18	93.80	94.28	96.99			
19				97.38	98.72	94.66

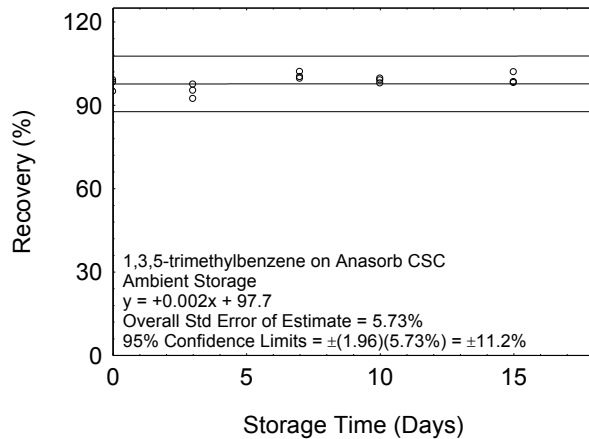


Figure 4.4.1.5. Ambient storage test for 1,3,5-trimethylbenzene on 226-01 Anasorb CSC.

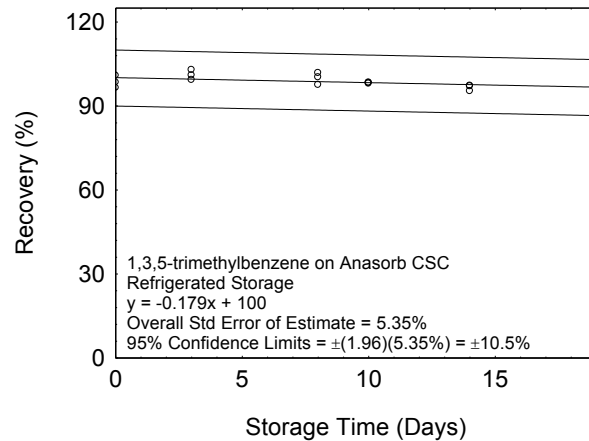


Figure 4.4.1.6. Refrigerated storage test for 1,3,5-trimethylbenzene on 226-01 Anasorb CSC.

4.4.2 SKC 575-002 Anasorb 747

Storage samples for 1,2,3-trimethylbenzene, 1,2,4-trimethylbenzene, and 1,3,5-trimethylbenzene were prepared by sampling a dynamically generated controlled test atmosphere using the recommended sampling time. The concentrations for ambient storage of 1,2,3-trimethylbenzene, 1,2,4-trimethylbenzene, and 1,3,5-trimethylbenzene in the test atmosphere were the target concentration (23.7, 25.3, 25.0 ppm, or 116.6, 124.4, 122.7 mg/m³) and the relative humidity was 65.8% at 24.7 °C. The concentrations for refrigerated storage of 1,2,3-trimethylbenzene, 1,2,4-trimethylbenzene, and 1,3,5-trimethylbenzene in the test atmosphere were the target concentration (23.8, 25.4, 25.1 ppm, or 117.1, 124.9, 123.2 mg/m³) and the relative humidity was 64.7% at 26.1 °C. Eighteen storage samples were prepared for each test. Three samples were analyzed on the day of generation (one refrigerated reference sample was lost in analysis). Fifteen samples were stored at reduced temperature (4 °C) and the other fifteen were stored in a closed drawer at ambient temperature (about 22 °C). At 3-4 day interval, three samples were selected from each of the two storage sets and analyzed. Sample results are not corrected for extraction efficiency.

Table 4.4.2.1
Storage Test for 1,2,3-Trimethylbenzene
on SKC 575-002 Anasorb 747

time (days)	ambient storage recovery (%)			refrigerated storage recovery (%)		
	0	93.67	96.19	95.60	88.35	91.90
3	90.25	90.62	87.23	79.40	85.46	83.38
7	95.55	93.07	100.5			
8				79.68	86.82	88.08
10	88.46	95.66	88.81	83.22	81.13	85.29
14				79.43	80.75	83.59
15	90.92	91.85	90.50			
18	94.86	89.26	90.17			
19				81.58	80.65	84.12

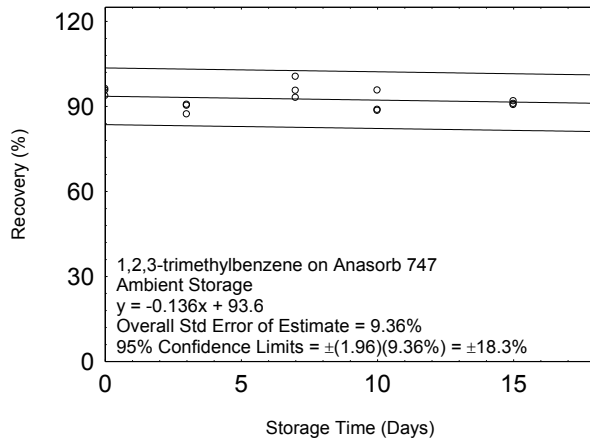


Figure 4.4.2.1. Ambient storage test for 1,2,3-trimethylbenzene on SKC 575-002 Anasorb 747.

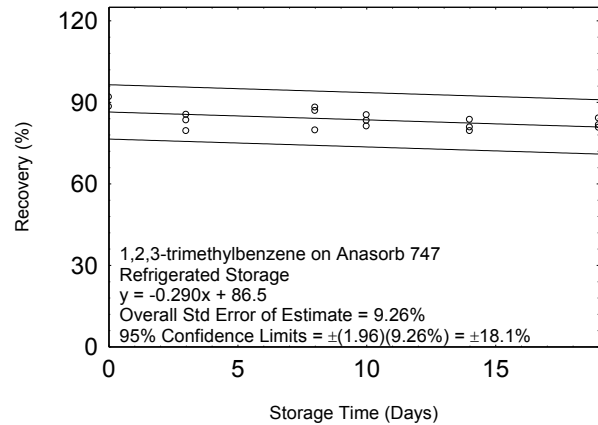


Figure 4.4.2.2. Refrigerated storage test for 1,2,3-trimethylbenzene on SKC 575-002 Anasorb 747.

Table 4.4.2.2
Storage Test for 1,2,4-Trimethylbenzene
on SKC 575-002 Anasorb 747

time (days)	ambient storage recovery (%)			refrigerated storage recovery (%)		
	0	101.3	103.0	102.1	95.47	99.06
3	95.92	96.37	93.47	86.62	92.48	90.71
7	100.7	98.99	104.7			
8				85.47	93.04	95.04
10	92.71	102.5	92.02	90.64	89.59	92.15
14				86.42	87.65	90.58
15	96.16	97.64	96.68			
18	101.3	95.95	96.75			
19				88.63	88.59	90.77

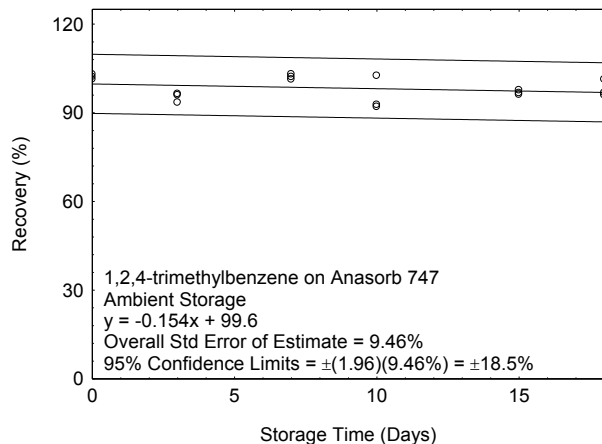


Figure 4.4.2.3. Ambient storage test for 1,2,4-trimethylbenzene on SKC 575-002 Anasorb 747.

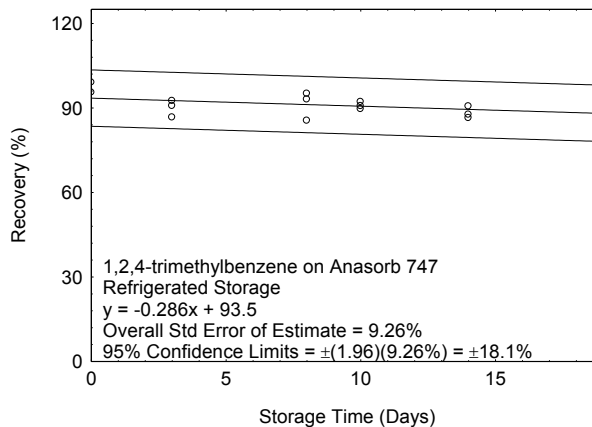


Figure 4.4.2.4. Refrigerated storage test for 1,2,4-trimethylbenzene on SKC 575-002 Anasorb 747.

Table 4.4.2.3
Storage Test for 1,3,5-Trimethylbenzene
on SKC 575-002 Anasorb 747

time (days)	ambient storage recovery (%)			refrigerated storage recovery (%)		
	0	103.8	105.7	104.7	98.47	102.1
3	97.07	98.22	94.98	88.53	94.46	93.55
7	102.2	100.9	105.3			
8				88.48	96.07	98.20
10	93.93	104.1	93.17	93.17	91.81	93.01
14				89.54	89.63	92.95
15	97.41	98.93	98.17			
18	103.0	96.64	98.31			
19				92.17	91.93	94.06

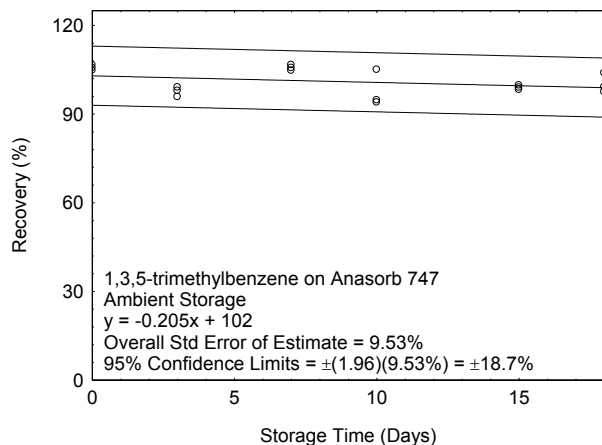


Figure 4.4.2.5. Ambient storage test for 1,3,5-trimethylbenzene on SKC 575-002 Anasorb 747.

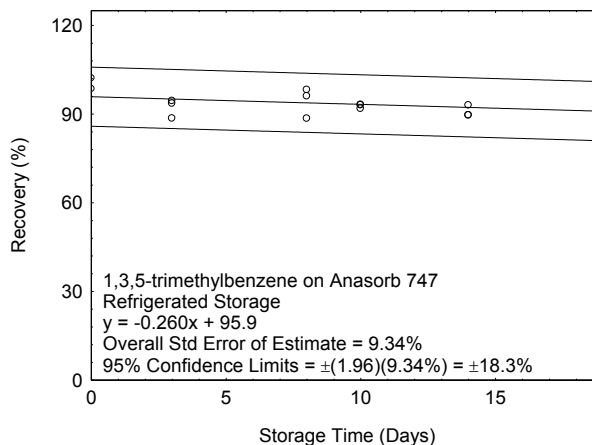


Figure 4.4.2.6. Refrigerated storage test for 1,3,5-trimethylbenzene on SKC 575-002 Anasorb 747.

4.5 Precision (overall procedure)

The precision of the overall procedure at the 95% confidence level is obtained by multiplying the overall standard error of estimate by 1.96 (the z-statistic from the standard normal distribution at the 95% confidence level). Ninety-five percent confidence intervals are drawn about the regression lines in the storage stability figures shown in Section 4.4.

4.5.1 226-01 Anasorb CSC

The precision of the overall procedure at the 95% confidence level for the 4 °C 19-day storage test (at the target concentration) for 1,2,3-trimethylbenzene, 1,2,4-trimethylbenzene, and 1,3,5-trimethylbenzene on Anasorb CSC are ± 10.9 , ± 10.7 , and $\pm 10.5\%$. It was obtained from the overall standard error of estimate (5.57%, 5.46%, and 5.35%) of the data shown in Figures 4.4.1.1-4.4.1.4. They contain an additional 5% for sampling pump error.

4.5.2 SKC 575-002 Anasorb 747

The precisions of the overall procedure at the 95% confidence level for the 4 °C 19-day storage test (at the target concentration) for 1,2,3-trimethylbenzene, 1,2,4-trimethylbenzene, and 1,3,5-trimethylbenzene on 575-002 Anasorb 747 are given in Table 4.5.2. They each include an additional $\pm 8.7\%$ for sampling rate variation.^{9,10} There are different values given, depending on whether both, either, or neither temperature (*T*) or atmospheric pressure (*P*) are known at the sampling site. If the sampling site temperature is unknown, it is assumed to be 22.2 ± 15 °C (72 ± 27 °F) and a variability of $\pm 7.7\%$ is included. If the atmospheric pressure is not known, it is estimated from the sampling site elevation and a variability of $\pm 3\%$ is included.

Table 4.5.2
Precision of the Overall Procedure for SKC 575-002 Anasorb 747

known condition	1,2,3-trimethylbenzene		1,2,4-trimethylbenzene		1,3,5-trimethylbenzene	
	error (%)	precision ($\pm\%$)	error (%)	precision ($\pm\%$)	error (%)	precision ($\pm\%$)
both T & P	9.26	18.1	9.26	18.1	9.34	18.3
only T	9.74	19.1	9.73	19.1	9.81	19.2
only P	12.0	23.5	12.0	23.5	12.1	23.7
neither T nor P	12.4	24.3	12.4	24.3	12.5	24.5

4.5.3 Recovery

The recovery of 1,2,3-trimethylbenzene, 1,2,4-trimethylbenzene, and 1,3,5-trimethylbenzene from samples used in a 19-day storage test remained above 87.66%, 92.31% and 96.60% when the samples were stored at 4 °C for 226-01 Anasorb CSC. The recovery of 1,2,3-trimethylbenzene, 1,2,4-trimethylbenzene, and 1,3,5-trimethylbenzene from samples used in a 19-day storage test remained above 80.99%, 88.07%, and 90.96% when the samples were stored at 4 °C for SKC 575-002 Anasorb 747. These results are not corrected for extraction efficiency.

4.6 Reproducibility

Six samples were prepared for both types of sampler by sampling a dynamically generated controlled test atmosphere similar to that used in the collection of the storage samples. The concentration of 1,2,3-trimethylbenzene, 1,2,4-trimethylbenzene, and 1,3,5-trimethylbenzene in the test atmosphere was the target concentration (23.9 ppm, 25.5 ppm, 25.2 ppm, or 117.6 mg/m³, 125.5 mg/m³, 123.8 mg/m³) and the relative humidity was 64.28% at 25.8 °C. The samples were submitted to the OSHA Salt Lake Technical Center for analysis. SKC 226-01

⁹ Hendricks, W. Development of a Protocol for Laboratory Testing of Diffusive Samplers, 1996. United States Department of Labor, Occupational Safety & Health Administration Web site. <http://osha.gov/dts/sltc/methods/studies/3movm/3movm.html> (accessed February 2016).

¹⁰ Hendricks, W. Determination of the Sampling Rate Variation for SKC 575 Series Passive Samplers, 1998. United States Department of Labor, Occupational Safety & Health Administration Web site. <http://osha.gov/dts/sltc/methods/studies/skc575/skc575.html> (accessed February 2016).

Anasorb CSC samples were stored for 5 days and SKC 575-002 Anasorb 747 were stored for 13 days at 4 °C before analysis. Sample results were corrected for extraction efficiency. No sample result for 1,2,3-trimethylbenzene, 1,2,4-trimethylbenzene, or 1,3,5-trimethylbenzene had a deviation greater than the precision of the overall procedure determined in Section 4.5. Theoretical masses for 575-002 Anasorb 747 were determined using the sampling rate determined in Section 4.7 converted to its equivalent sampling rate at sampling site temperature and atmospheric pressure.

Table 4.6.1
Reproducibility Data for 1,2,3-trimethylbenzene
Collected on 226-01 Anasorb CSC

theoretical (µg/sample)	recovered (µg/sample)	recovery (%)	deviation (%)
1421.6	1400.6	98.52	-1.48
1439.1	1372.3	95.36	-4.64
1482.3	1440.9	97.20	-2.79
1442.9	1395.2	96.69	-3.31
1461.8	1414.3	96.75	-3.25
1423.0	1373.1	96.49	-3.51

Table 4.6.2
Reproducibility Data for 1,2,3-trimethylbenzene
Collected on SKC 575-002 Anasorb 747

theoretical (µg/sample)	recovered (µg/sample)	recovery (%)	deviation (%)
381.7	360.4	94.42	-5.58
381.7	371.7	97.38	-2.62
381.7	366.4	95.99	-4.01
381.7	365.9	95.86	-4.14
381.7	359.5	94.18	-5.82
381.7	344.1	90.15	-9.85

Table 4.6.3
Reproducibility Data for 1,2,4-trimethylbenzene
Collected on 226-01 Anasorb CSC

theoretical (µg/sample)	recovered (µg/sample)	recovery (%)	deviation (%)
1516.8	1615.5	106.5	+6.50
1535.5	1585.8	103.3	+3.30
1581.6	1664.3	105.2	+5.20
1539.5	1616.0	105.0	+5.00
1559.7	1628.7	104.4	+4.40
1518.3	1583.6	104.3	+4.30

Table 4.6.4
Reproducibility Data for 1,2,4-trimethylbenzene
Collected on SKC 575-002 Anasorb 747

theoretical (µg/sample)	recovered (µg/sample)	recovery (%)	deviation (%)
410.7	411.2	100.1	+0.10
410.7	422.2	102.8	+2.80
410.7	417.0	101.5	+1.50
410.7	417.0	101.5	+1.50
410.7	410.6	99.98	-0.02
410.7	388.4	94.57	-5.43

Table 4.6.5
Reproducibility Data for 1,3,5-trimethylbenzene
Collected on 226-01 Anasorb CSC

theoretical (µg/sample)	recovered (µg/sample)	recovery (%)	deviation (%)
1496.0	1570.0	104.9	+4.90
1514.4	1545.1	102.0	+2.00
1559.9	1621.1	103.9	+3.90
1518.5	1577.0	103.9	+3.90
1538.4	1580.5	102.7	+2.70
1497.5	1538.7	102.8	+2.80

Table 4.6.6
Reproducibility Data for 1,3,5-trimethylbenzene
Collected on SKC 575-002 Anasorb 747

theoretical (µg/sample)	recovered (µg/sample)	recovery (%)	deviation (%)
418.9	410.9	98.09	-1.91
418.9	420.6	100.4	+0.40
418.9	416.5	99.43	-0.57
418.9	416.9	99.52	-0.48
418.9	409.2	97.68	-2.32
418.9	385.2	91.96	-8.04

4.7 Sampler capacity and sampling rate for 226-01 Anasorb CSC and SKC 575-002 Anasorb 747

4.7.1 226-01 Anasorb CSC

The sampling capacity of the front section of an coconut-shell charcoal tube was tested by sampling a dynamically generated controlled test atmosphere containing 1,2,3-trimethylbenzene at two times the target concentration (41.5 ppm or 204 mg/m³), 1,2,4-trimethylbenzene at two times the target concentration (49.9 ppm or 245 mg/m³), 1,3,5-trimethylbenzene at two times the target concentration (49.2 ppm or 242 mg/m³) with a 64.6% relative humidity at 27.5 °C. The samples were collected at 50 mL/min. A second tube was placed behind the first tube and changed at 375, 452, and 600 minutes. No analyte was observed on any of the second tubes nor was any analyte

observed on the back-up sections of the first tube. The recommended sampling time is 4 hours.

4.7.2 SKC 575-002 Anasorb 747

The sampling rate and sampler capacity for SKC 575-002 Anasorb 747 were determined by sampling a dynamically generated controlled test atmosphere for increasing time intervals. Sampler capacity is exceeded when the plotted sampling rate decreases rapidly as the sampler becomes saturated. The concentration of the test atmosphere was the same as in section 4.7.1. The preliminary sampling rate was determined by averaging the nine values for the 0.5, 1 and 2 hour samples. Horizontal lines were placed 10% above and 10% below the preliminary sampling rate. The sampling rates for 1,2,3-trimethylbenzene, 1,2,4-trimethylbenzene, and 1,3,5-trimethylbenzene are 11.6, 11.7, and 12.1 mL/min at 760 mmHg and 25 °C and represent the average of all values between the lines. The standard deviation and RSD are 0.32, 0.41, 0.36 mL/min and 2.75, 3.50, 2.99%, respectively. The data obtained are shown in Tables 4.7.2.1-4.7.2.3 and Figure 4.7.2.1-4.7.2.3. Mass collected is corrected for extraction efficiency. The recommended sampling time is 4 hours.

Table 4.7.2.1
Determination of Sampling Rate and Time
for 1,2,3-Trimethylbenzene

time (h)	sampling rate (mL/min)		
	First	second	third
0.083	12.0	12.0	11.9
0.167	11.1	11.6	11.2
0.5	11.6	11.4	11.3
1	11.8	11.6	11.4
2	11.3	11.4	11.6
3	11.4	11.4	11.8
4	11.2	11.5	11.5
6	11.6	11.4	11.5
8	11.8	11.6	12.6
10	11.2	12.0	12.9

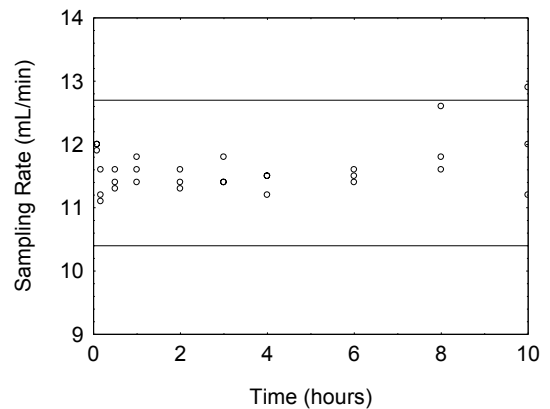


Figure 4.7.2.1. Plot of data to determine the sampling rate for 1,2,3-trimethylbenzene.

Table 4.7.2.2
Determination of Sampling Rate and Time
for 1,2,4-Trimethylbenzene

time (h)	sampling rate (mL/min)		
	First	second	third
0.083	11.0	11.7	10.6
0.167	11.2	11.1	10.5
0.5	11.7	11.6	11.5
1	12.1	11.8	11.8
2	11.6	11.8	12.0
3	11.7	11.7	12.0
4	11.4	11.8	11.8
6	11.8	11.8	12.0
8	12.2	11.8	12.7
10	11.6	12.3	13.2

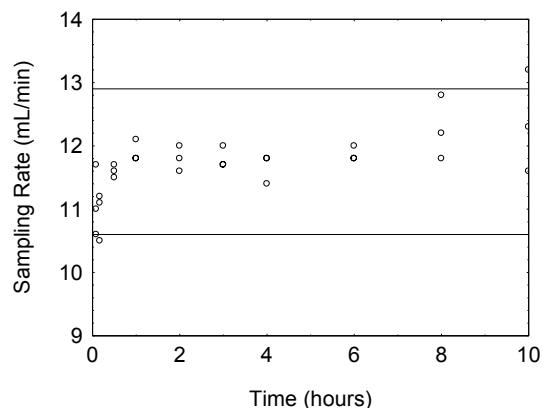


Figure 4.7.2.2. Plot of data to determine the sampling rate for 1,2,4-trimethylbenzene.

Table 4.7.2.3
Determination of Sampling Rate and Time
for 1,3,5-Trimethylbenzene

time (h)	sampling rate (mL/min)		
	First	second	third
0.083	12.1	11.8	12.5
0.167	12.0	12.0	11.9
0.5	12.4	12.3	11.6
1	12.3	11.9	12.0
2	11.8	12.0	12.2
3	11.9	11.7	12.0
4	11.4	12.0	12.0
6	11.8	11.9	12.2
8	12.4	11.8	12.7
10	11.6	12.4	13.2

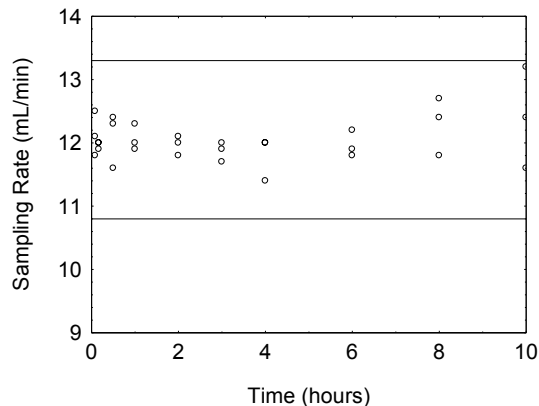


Figure 4.7.2.3. Plot of data to determine the sampling rate for 1,3,5-trimethylbenzene.

4.8 Extraction efficiency and stability of extracted samples

The extraction efficiency is affected by the extraction solvent, the internal standard, the sampling medium, and the technique used to extract the samples. Other reagents and techniques than described in this method can be used provided they are tested as specified in the validation guidelines.¹¹

4.8.1 226-01 Anasorb CSC

Extraction efficiency

The extraction efficiency of 1,2,3-trimethylbenzene, 1,2,4-trimethylbenzene, and 1,3,5-trimethylbenzene was determined by liquid-spiking four coconut-shell charcoal tubes at each concentration level. These samples were stored overnight at ambient temperature and then analyzed. The overall mean extraction efficiency over the working range of 0.1 to 2 times the target concentration was 92.94% for 1,2,3-trimethylbenzene, 96.97% for 1,2,4-trimethylbenzene, and 98.97% for 1,3,5-trimethylbenzene. The extraction efficiency at the RQL was 96.48% for 1,2,3-trimethylbenzene, 92.42% for 1,2,4-trimethylbenzene, and 95.87% for 1,3,5-trimethylbenzene. The presence of water had no significant effect on extraction efficiency. The extraction efficiencies for the RQL and for the wet samplers are not included in the overall mean. Wet media were prepared by drawing humid air (62.3% relative humidity at 26.4 °C) at 50 mL/min for 4 hours. The data obtained are shown in Table 4.8.1.1-4.8.1.3.

¹¹ Eide, M.; Simmons, M.; Hendricks, W. Validation Guidelines for Air Sampling Methods Utilizing Chromatographic Analysis, 2010. United States Department of Labor, Occupational Safety & Health Administration Web site. <http://www.osha.gov/dts/sltc/methods/chromguide/chromguide.pdf> (accessed March 2016).

Table 4.8.1.1
Extraction Efficiency of 1,2,3-Trimethylbenzene from 226-01 Anasorb CSC

<u>level</u>		<u>sample number</u>				
× target concn	µg per sample	1	2	3	4	mean
0.1	95.30	91.83	92.59	92.54	90.02	91.75
0.25	363.4	90.11	93.03	94.89	90.97	92.25
0.5	710.4	92.53	92.05	91.93	91.51	92.01
1.0	1272	94.57	94.57	94.39	94.07	94.40
1.5	1976	92.79	93.88	93.49	93.10	93.32
2.0	2667	94.08	94.35	93.78	93.47	93.92
RQL	5.91	96.50	94.82	103.4	91.19	96.48
1.0 (wet)	1302	85.01	89.45	92.32	94.17	90.24

Table 4.8.1.2
Extraction Efficiency of 1,2,4-Trimethylbenzene from 226-01 Anasorb CSC

<u>level</u>		<u>sample number</u>				
× target concn	µg per sample	1	2	3	4	mean
0.1	141.1	94.45	94.99	95.94	96.58	95.49
0.25	369.2	96.95	96.14	95.09	97.25	96.36
0.5	732.0	97.92	97.37	97.47	97.15	97.48
1.0	1389	98.49	97.44	97.04	97.90	97.72
1.5	2220	97.25	97.55	98.59	96.96	97.59
2.0	2916	96.57	97.75	97.14	97.30	97.19
RQL	6.40	93.41	92.91	89.58	93.78	92.42
1.0 (wet)	1395	86.29	93.60	96.39	97.12	93.35

Table 4.8.1.3
Extraction Efficiency of 1,3,5-Trimethylbenzene from 226-01 Anasorb CSC

<u>level</u>		<u>sample number</u>				
× target concn	µg per sample	1	2	3	4	mean
0.1	151.8	98.72	98.42	97.50	99.58	98.56
0.25	349.0	99.45	98.32	100.0	98.24	99.00
0.5	718.1	98.93	99.13	95.18	99.07	98.08
1.0	1396	99.81	99.19	99.29	99.86	99.54
1.5	2125	99.03	99.81	100.1	99.71	99.66
2.0	2921	98.04	99.05	99.21	99.63	98.98
RQL	6.41	95.21	96.13	95.99	96.14	95.87
1.0 (wet)	1355	98.39	98.58	97.27	99.09	98.33

4.8.2 SKC 575-002 Anasorb 747

Extraction efficiency

The extraction efficiency of 1,2,3-trimethylbenzene, 1,2,4-trimethylbenzene, and 1,3,5-trimethylbenzene was determined by liquid-spiking four SKC Anasorb 747 badges at each concentration level. These samples were stored overnight at ambient temperature and then analyzed. The overall mean extraction efficiency over the working range of 0.1 to 2 times the target concentration was 88.67% for 1,2,3-trimethylbenzene, 92.70% for 1,2,4-trimethylbenzene, and 97.53% for 1,3,5-trimethylbenzene. The extraction efficiency at the RQL was 85.08% for 1,2,3-trimethylbenzene, 105.0% for 1,2,4-trimethylbenzene, and 97.16% for 1,3,5-trimethylbenzene. The presence of water had no significant effect on extraction

efficiency. The extraction efficiencies for the RQL and for the wet samplers are not included in the overall mean. Wet media were prepared by drawing humid air (62.3% relative humidity at 26.4 °C) for 4 hours. The data obtained are shown in Table 4.8.2.1-4.8.2.3.

Table 4.8.2.1
Extraction Efficiency of 1,2,3-Trimethylbenzene
from SKC 575-002 Anasorb 747

<u>level</u>		<u>sample number</u>				
× target concn	µg per sample	1	2	3	4	mean
0.1	35.39	87.77	89.13	90.07	83.13	87.53
0.25	104.4	88.69	87.80	86.30	85.02	86.95
0.5	200.1	86.07	87.68	86.28	88.29	87.08
1.0	351.2	90.54	90.74	90.74	91.88	90.98
1.5	544.1	89.18	91.39	89.63	90.61	90.20
2.0	738.3	88.36	89.22	90.55	88.87	89.25
RQL	10.8	84.33	91.16	83.53	81.28	85.08
1.0 (wet)	262.0	83.55	84.18	83.64	82.02	88.35

Table 4.8.2.2
Extraction Efficiency of 1,2,4-Trimethylbenzene
from SKC 575-002 Anasorb 747

<u>level</u>		<u>sample number</u>				
× target concn	µg per sample	1	2	3	4	mean
0.1	33.92	86.81	90.25	90.76	90.51	89.58
0.25	101.5	92.03	91.10	89.91	94.25	91.82
0.5	201.6	94.82	92.99	94.33	93.35	93.87
1.0	376.5	91.65	94.85	91.78	93.38	92.92
1.5	579.9	92.36	93.96	94.84	95.08	94.06
2.0	782.4	92.68	94.82	93.42	94.82	93.94
RQL	11.2	105.3	107.5	104.1	103.0	105.0
1.0 (wet)	272.1	87.45	90.09	90.95	89.87	89.59

Table 4.8.2.3
Extraction Efficiency of 1,3,5-Trimethylbenzene
from SKC 575-002 Anasorb 747

<u>level</u>		<u>sample number</u>				
× target concn	µg per sample	1	2	3	4	mean
0.1	34.89	94.35	98.54	100.2	95.19	97.06
0.25	104.1	95.41	97.41	95.14	98.43	96.60
0.5	209.1	96.69	97.65	97.13	97.18	97.16
1.0	389.3	96.00	97.46	96.97	97.32	96.94
1.5	594.8	97.21	99.10	98.13	98.73	98.29
2.0	795.5	98.90	99.07	99.36	99.23	99.14
RQL	11.2	95.22	101.7	97.2	94.53	97.16
1.0 (wet)	272.2	94.14	95.34	95.23	95.83	95.14

4.8.3 Stability of extracted samples

The stability of extracted samples was examined by reanalyzing the target concentration samples 24, 48, and 72 hours after the initial analysis. After the original analysis was performed two vials were recapped with new septa which were replaced

after each reanalysis. The remaining two vials retained their punctured septa throughout the test. All samples were allowed to stand in the autosampler tray at 22.2 °C. The samples were reanalyzed with freshly prepared standards. Diff is the difference between the initial analysis and the subsequent analysis. Each septum was punctured four times for each injection. The data obtained are shown in Table 4.8.3.1-4.8.3.6..

Table 4.8.3.1
Stability of Extracted Samples on 226-01 Anasorb CSC for 1,2,3-Trimethylbenzene

<u>punctured septa replaced</u>							<u>punctured septa retained</u>						
initial (%)	24 h (%)	diff (%)	48 h (%)	diff (%)	72 h (%)	Diff (%)	initial (%)	24 h (%)	diff (%)	48 h (%)	diff (%)	72 h (%)	diff (%)
103.6	101.8	-1.8	100.0	-3.6	100.5	-3.1	106.4	104.4	-2.0	101.5	-4.9	101.7	-4.7
106.7	105.3	-1.4	102.7	-4.0	103.2	-3.5	106.7	104.2	-2.5	101.6	-5.1	101.6	-5.1
			mean							mean			
105.2	103.6	-1.6	101.4	-3.8	101.9	-3.3	106.6	104.3	-2.3	101.6	-5.0	101.7	-4.9

Table 4.8.3.2
Stability of Extracted Samples on 226-01 Anasorb CSC for 1,2,4-Trimethylbenzene

<u>punctured septa replaced</u>							<u>punctured septa retained</u>						
initial (%)	24 h (%)	diff (%)	48 h (%)	diff (%)	72 h (%)	diff (%)	initial (%)	24 h (%)	diff (%)	48 h (%)	diff (%)	72 h (%)	diff (%)
102.3	101.4	-0.9	99.23	-3.1	99.65	-2.7	105.2	104.2	-1.0	101.2	-4.0	101.6	-3.6
105.3	104.7	-0.6	101.8	-3.5	102.3	-3.0	105.3	103.4	-1.9	101.6	-3.7	101.0	-4.3
			mean							mean			
103.8	103.1	-0.7	100.5	-3.3	101.0	-2.8	105.3	103.8	-1.5	101.4	-3.9	101.3	-4.0

Table 4.8.3.3
Stability of Extracted Samples on 226-01 Anasorb CSC for 1,3,5-Trimethylbenzene

<u>punctured septa replaced</u>							<u>punctured septa retained</u>						
initial (%)	24 h (%)	diff (%)	48 h (%)	diff (%)	72 h (%)	diff (%)	initial (%)	24 h (%)	diff (%)	48 h (%)	diff (%)	72 h (%)	diff (%)
102.7	102.2	-0.5	99.95	-2.8	100.3	-2.4	105.8	105.4	-0.4	102.4	-3.4	102.9	-2.9
105.9	105.6	-0.3	102.5	-3.4	103.1	-2.8	105.7	104.3	-1.4	101.9	-3.8	102.1	-3.6
			mean							mean			
104.3	103.9	-0.4	101.2	-3.1	101.7	-2.6	105.8	104.9	-0.9	102.2	-3.6	102.5	-3.3

Table 4.8.3.4
Stability of Extracted Samples on SKC 575-002 Anasorb 747 for 1,2,3-Trimethylbenzene

<u>punctured septa replaced</u>							<u>punctured septa retained</u>						
initial (%)	24 h (%)	diff (%)	48 h (%)	diff (%)	72 h (%)	diff (%)	initial (%)	24 h (%)	Diff (%)	48 h (%)	diff (%)	72 h (%)	diff (%)
102.7	105.3	+2.6	105.2	+2.5	106.3	+3.6	101.8	104.7	+2.9	104.6	+2.8	105.4	+3.6
100.2	102.1	+1.9	102.1	+1.9	103.6	+3.4	103.9	106.4	+2.5	106.2	+2.3	107.3	+3.4
			mean							mean			
101.5	103.7	+2.2	103.7	+2.2	105.0	+3.5	102.8	105.6	+2.7	105.4	+2.6	106.4	+3.5

Table 4.8.3.5
Stability of Extracted Samples on SKC 575-002 Anasorb 747 for 1,2,4-Trimethylbenzene

<u>punctured septa replaced</u>							<u>punctured septa retained</u>						
initial (%)	24 h (%)	diff (%)	48 h (%)	diff (%)	72 h (%)	diff (%)	initial (%)	24 h (%)	diff (%)	48 h (%)	diff (%)	72 h (%)	diff (%)
106.1	108.7	+2.6	108.5	+2.4	109.7	+3.6	106.0	108.2	+2.2	108.8	+2.8	109.6	+3.6
104.4	106.3	+1.9	106.9	+2.5	108.1	+3.7	107.3	110.3	+3.0	109.8	+2.5	110.7	+3.4
			mean							mean			
105.3	107.5	+2.2	107.7	+2.4	108.9	+3.6	106.7	109.3	+2.6	109.3	+2.6	110.2	+3.5

Table 4.8.3.6
Stability of Extracted Samples on SKC 575-002 Anasorb 747 for 1,3,5-Trimethylbenzene

<u>punctured septa replaced</u>							<u>punctured septa retained</u>						
initial (%)	24 h (%)	diff (%)	48 h (%)	diff (%)	72 h (%)	diff (%)	initial (%)	24 h (%)	diff (%)	48 h (%)	diff (%)	72 h (%)	diff (%)
108.3	111.4	+3.1	111.1	+2.8	112.1	+3.8	108.9	111.8	+2.9	111.9	+3.0	112.4	+3.5
107.9	110.1	+2.2	110.3	+2.4	111.0	+3.1	109.0	112.6	+3.6	112.4	+3.4	113.1	+4.1
			mean							mean			
108.1	110.8	+2.7	110.7	+2.6	111.6	+3.5	109.0	112.2	+3.2	112.2	+3.2	112.8	+3.8

4.9 Sampling interferences

The tested sampling interferences had no significant effect on the ability of 226-01 Anasorb CSC and 575-002 Anasorb 747 samplers to collect or retain trimethylbenzene isomers.

4.9.1 226-01 Anasorb CSC

Retention

Retention was tested by sampling a dynamically generated controlled test atmosphere containing two times the target concentration (49.5, 52.9, 52.1 ppm or 243, 260, 256 mg/m³) of 1,2,3-trimethylbenzene, 1,2,4-trimethylbenzene, and 1,3,5-trimethylbenzene at 61.8% relative humidity and 26.2 °C. The test atmosphere was sampled with six Anasorb CSC tubes at 50 mL/min for 60 min. Sampling was discontinued and the samplers were separated into two sets of 3 samplers each. The generation system was flushed with contaminant-free air. Contaminant-free air is laboratory conditioned air at known relative humidity and temperature but without any added chemical except water. Sampling was resumed with the second set of three samples and contaminant-free air at 61.4% relative humidity and 25.9 °C at 50 mL/min for 180 min and then all six samplers were analyzed. The data obtained are shown in Table 4.9.1.1.

Table 4.9.1.1
Retention of Trimethylbenzene Isomers
on 226-01 Anasorb CSC

set	1,2,3-trimethylbenzene				1,2,4-trimethylbenzene				1,3,5-trimethylbenzene			
	1	2	3	mean	1	2	3	mean	1	2	3	mean
first	107.5	103.3	103.5	104.8	106.8	102.9	103.0	104.2	107.6	103.5	103.9	105.0
second	98.48	99.49	100.4	99.46	97.87	99.15	97.00	98.01	98.50	98.87	101.1	99.49
second/first				94.93				94.03				94.75

Low humidity

The effect of low humidity was tested by sampling a dynamically generated controlled test atmosphere containing two times the target concentration at 16.5% relative humidity and 26.3 °C. The test atmosphere was sampled with three charcoal tubes at 50 mL/min for 240 min. All of the samples were immediately analyzed. The data obtained are shown in Table 4.9.1.2.

Table 4.9.1.2
Recovery of Trimethylbenzene Isomers with Low Humidity
on 226-01 Anasorb CSC

analyte	target	recovery %		
		sample 1	sample 2	sample 3
1,2,3-trimethylbenzene	47.6 ppm (234 mg/m ³)	101.6	102.9	100.4
1,2,4-trimethylbenzene	50.9 ppm (250 mg/m ³)	100.3	101.5	98.94
1,3,5-trimethylbenzene	50.1 ppm (246 mg/m ³)	100.8	101.9	102.7

Low concentration

The effect of low concentration was tested by sampling a dynamically generated controlled test atmosphere containing 0.1 times the target concentration of 1,2,3-trimethylbenzene, 1,2,4-trimethylbenzene, and 1,3,5-trimethylbenzene at 60.8% relative humidity and 26.2 °C. The test atmosphere was sampled with three charcoal tubes at 50 mL/min for 240 min. All of the samples were immediately analyzed. The data obtained are shown in Table 4.9.1.3.

Table 4.9.1.3
Recovery of Trimethylbenzene Isomers with Low Concentration
on 226-01 Anasorb CSC

analyte	target	recovery %		
		sample 1	sample 2	sample 3
1,2,3-trimethylbenzene	2.36 ppm (11.6 mg/m ³)	101.4	99.42	98.37
1,2,4-trimethylbenzene	2.52 ppm (12.4 mg/m ³)	101.8	98.38	98.64
1,3,5-trimethylbenzene	2.48 ppm (12.2 mg/m ³)	101.8	100.0	100.1

Chemical interference

The effect of potential chemical sampling interference was tested by sampling a dynamically generated controlled test atmosphere containing one times the target concentration of 1,2,3-trimethylbenzene, 1,2,4-trimethylbenzene, and 1,3,5-trimethylbenzene at 62.5% relative humidity and 26.4 °C and toluene at 276.3 ppm or 1041 mg/m³. The test atmosphere was sampled with three charcoal tubes at 50 mL/min for 240 min. All of the samples were immediately analyzed. The data obtained are shown in Table 4.9.1.4.

Table 4.9.1.4
Recovery of Trimethylbenzene Isomers with a Toluene Interference
on 226-01 Anasorb CSC

analyte	target	recovery %		
		sample 1	sample 2	sample 3
1,2,3-trimethylbenzene	23.7 ppm (116.7 mg/m ³)	99.34	101.2	98.97
1,2,4-trimethylbenzene	25.3 ppm (124.5 mg/m ³)	98.67	100.5	98.36
1,3,5-trimethylbenzene	25.0 ppm (122.8 mg/m ³)	98.87	100.7	98.77

4.9.2 SKC 575-002 Anasorb 747

Reverse diffusion

Reverse diffusion was tested by sampling a dynamically generated controlled test atmosphere containing two times the target concentration (49.5, 52.9, 52.1 ppm or 243, 260, 256 mg/m³) of 1,2,3-trimethylbenzene, 1,2,4-trimethylbenzene, and 1,3,5-trimethylbenzene at 61.8% relative humidity and 26.2 °C. The test atmosphere was sampled with six SKC 575-002 Anasorb 747 Passive Samplers for 60 min. Sampling was discontinued and the samplers were separated into two sets of 3 samplers each. The generation system was flushed with contaminant-free air. Contaminant-free air is

laboratory conditioned air at known relative humidity and temperature but without any added chemical except water. Sampling was resumed with the second set of three samples and contaminant-free air at 61.4% relative humidity and 25.87 °C for 180 min and then all six samplers were analyzed. The data obtained are shown in Table 4.9.2.1.

Table 4.9.2.1
Reverse Diffusion of Trimethylbenzene Isomers
on SKC 575-002 Anasorb 747

set	1,2,3-trimethylbenzene				1,2,4-trimethylbenzene				1,3,5-trimethylbenzene			
	1	2	3	mean	1	2	3	mean	1	2	3	mean
first	93.38	92.43	91.51	92.44	101.3	100.5	100.1	100.6	98.22	97.69	96.67	97.53
second	96.28	93.92	94.12	94.77	105.4	104.6	101.2	103.7	105.6	104.8	101.4	103.9
second/first	102.5				103.1				106.6			

Low humidity

The effect of low humidity was tested by sampling a dynamically generated controlled test atmosphere containing two times the target concentration at 16.5% relative humidity and 26.3 °C. The test atmosphere was sampled with three passive samplers for 240 min. All of the samples were immediately analyzed. The data obtained are shown in Table 4.9.2.2.

Table 4.9.2.2
Recovery of Trimethylbenzene Isomers with Low Humidity
on SKC 575-002 Anasorb 747

analyte	target	recovery %		
		sample 1	sample 2	sample 3
1,2,3-trimethylbenzene	47.6 ppm (234 mg/m ³)	98.41	98.60	98.99
1,2,4-trimethylbenzene	50.8 ppm (250 mg/m ³)	100.5	101.6	101.7
1,3,5-trimethylbenzene	50.1 ppm (246 mg/m ³)	98.19	99.02	99.22

Low concentration

The effect of low concentration was tested by sampling a dynamically generated controlled test atmosphere containing 0.1 times the target concentration of 1,2,3-trimethylbenzene, 1,2,4-trimethylbenzene, and 1,3,5-trimethylbenzene at 60.8% relative humidity and 26.2 °C. The test atmosphere was sampled with three passive samplers for 240 min. All of the samples were immediately analyzed.

Table 4.9.2.3
Recovery of Trimethylbenzene Isomers with Low Concentration
on SKC 575-002 Anasorb 747

analyte	target	recovery %		
		sample 1	sample 2	sample 3
1,2,3-trimethylbenzene	2.36 ppm (11.6 mg/m ³)	97.82	93.07	93.03
1,2,4-trimethylbenzene	2.52 ppm (12.4 mg/m ³)	99.82	92.05	90.37
1,3,5-trimethylbenzene	2.48 ppm (12.2 mg/m ³)	96.50	94.62	92.44

Chemical sampling interference

The effect of potential chemical sampling interference was tested by sampling a dynamically generated controlled test atmosphere containing one times the target concentration of 1,2,3-trimethylbenzene, 1,2,4-trimethylbenzene, and 1,3,5-trimethylbenzene at 62.5% relative humidity and 26.4 °C and toluene at 276.3 ppm or 1041 mg/m³. The test atmosphere was sampled with three 575-002 passive samplers for 240 min. All of the samples were immediately analyzed.

Table 4.9.2.4
Recovery of Trimethylbenzene Isomers with a Toluene Interference
on SKC 575-002 Anasorb 747

analyte	target	recovery %		
		sample 1	sample 2	sample 3
1,2,3-trimethylbenzene	23.7 ppm (116.7 mg/m ³)	102.2	101.7	99.65
1,2,4-trimethylbenzene	25.3 ppm (124.5 mg/m ³)	104.7	104.4	102.5
1,3,5-trimethylbenzene	25.0 ppm (122.8 mg/m ³)	102.5	102.0	99.90

4.10 Qualitative analysis

When necessary, the identity or purity of an analyte peak can be confirmed by GC/MS or by another analytical procedure. For the levels analyzed in this method the use of GC/MS is recommended. A total ion chromatograph (TIC) and mass spectrum, obtained using the analytical conditions described below, are shown in Figure 4.10 and 4.11.

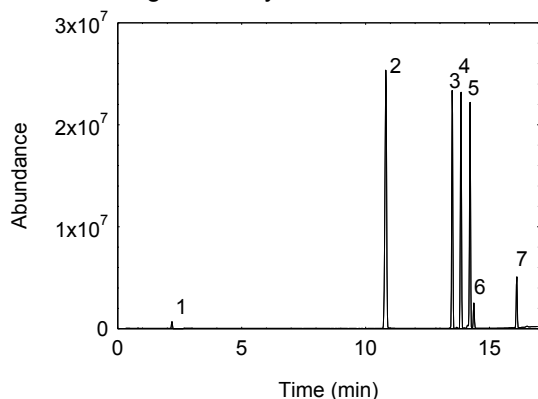


Figure 4.10.1. TIC of trimethylbenzene isomers. (1:CS₂; 2: DMF; 3: 1,3,5-trimethylbenzene; 4: 1,2,4-trimethylbenzene; 5: 1,2,3-trimethylbenzene; 6: interference from 1,2,3-trimethylbenzene; and 7: n-hexylbenzene (ISTD)).

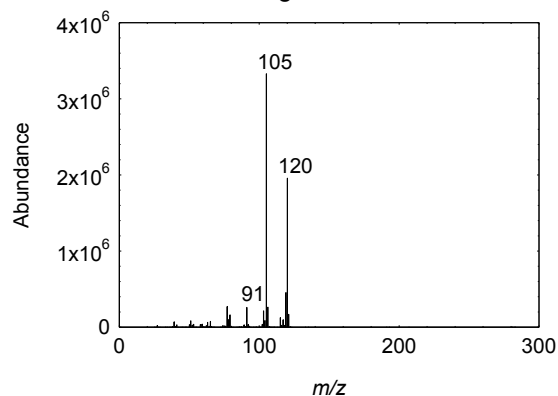


Figure 4.10.2. Mass spectrum of 1,3,5-trimethylbenzene in the extraction solvent.

GC conditions

oven temperature:	40 °C (hold 3 min), ramp to 90 °C at 6 °C/min (hold 0 min), ramp to 225 °C at 25 °C/min (hold 0.27 min)
injector temperature:	250 °C
run time:	17 min
column:	Agilent J&W DB-624, 30-m × 0.18-mm i.d., (1-μm df), (catalog no. 121-1324, or equivalent)
column mode:	constant flow (initial avg. velocity 44 cm/sec)
initial column gas flow:	1.5 mL/min (helium)
septum purge:	3.0 mL/min (helium)
injection size:	1.0 μL (150 to 1 split)
inlet liner:	Restek Sky 4.0 mm ID Low Pressure Drop Precision Inlet Liner w/wool (Restek catalog no. 23309.1, or equivalent)
retention times:	2.20 min (CS ₂) 10.8 min (DMF) 13.5 min (1,3,5-trimethylbenzene) 13.9 min (1,2,4-trimethylbenzene) 14.2 min (1,2,3-trimethylbenzene) 16.1 min (n-hexylbenzene ISTD)

MS conditions

mode:	EI
solvent delay:	0.2 min
timed events:	2.2 min MS off 2.6 min MS On
EMV mode:	gain factor 5
MS source:	250 °C
MS quad:	150 °C
MSD transfer line:	300 °C
scan parameters:	25-400 amu
threshold:	150

4.11 Generation of test atmospheres

A test atmosphere generator, as diagramed in Figure 4.11, was set up in a walk-in hood. House air was regulated using a Miller Nelson Model 401 Flow-Temperature-Humidity Control System. A measured flow of a mixture of 1:1:1 (v/v) of 1,2,3-trimethylbenzene, 1,2,4-trimethylbenzene, and 1,3,5-trimethylbenzene was introduced with an ISCO model 100DM syringe pump through a short length of 0.53-mm uncoated fused silica capillary tubing into a vapor generator where it was heated and evaporated into a measured flow of dilution air coming from the Miller Nelson control system. The trimethylbenzenes and dilution air flowed into a mixing chamber (76-cm × 15-cm) and then into a sampling chamber (56-cm × 9.5-cm). Samples were collected through sampling ports on the sampling chamber. Temperature and humidity were measured near the exit of the sampling chamber using a Vaisala HUMICAP® Hand-Held Humidity and Temperature Meter HM70.

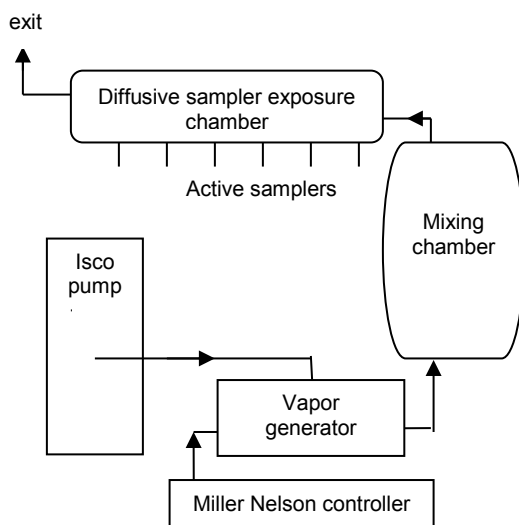


Figure 4.11. The test atmosphere generation and sample collection apparatus.